

**Appendix G**  
**Tank V-9 Analytical Sample Results Report**



## **Appendix G**

### **Tank V-9 Analytical Results Report**

#### **G-1. INTRODUCTION AND BACKGROUND**

This report presents the analytical results for samples collected from one of the remediation sites addressed under this document—the *Comprehensive Remedial Design/Remedial Action Work Plan for the Test Area North, Waste Area Group 1, Operable Unit 1-10, Group 2 Sites*—specifically Tank V-9, which is part of Technical Support Facility-18 (TSF-18). TSF-09 (Tanks V-1, V-2, and V-3) and TSF-18 (Tank V-9), the V-Tanks, are situated in an open area east of Test Area North-616 (TAN-616) and north of TAN-607. Waste was transferred from the TAN-616 evaporator pit sump and pump room sump, the TAN-607 laboratory drain, the TAN-607 Warm/Hot Shop drain, and TSF-21 (Valve Pit No. 2) through the TAN-1704 valve pit to Tank V-9. The overflow from Tank V-9 drained to the TSF-09 tanks (Idaho National Engineering and Environmental Laboratory [INEEL] 2001).

Tank V-9 was sampled from April 30 to May 8, 2001. Eight samples and one duplicate—for a total of nine samples—were collected for analysis. The primary objective of this sampling effort was to provide isotopic analyses that would support the Tank V-9 criticality evaluation. In addition to the uranium-234/235/238 analysis, the samples were also analyzed for percent moisture, bulk density, and toxicity characteristic leaching procedure (TCLP) metals. Additional isotopic uranium analyses were performed in August 2001, and the results are included in this report. The following sections provide brief descriptions of Tank v-9 and previous characterization efforts.

##### **G-1.1 TSF-18, Contaminated Tank southeast of Tank V-3**

TSF-18 includes a single conical-shaped sump tank (Tank V-9), tank contents, an aboveground sand filter, ancillary piping immediately in the vicinity of the tank, and surrounding contaminated soil. The abandoned underground storage tank is located in the open area between the TAN-616 and TAN-633 buildings and is adjacent to the southeast corner of TSF-09.

Tank V-9 was installed in 1953 as part of the V-Tank radioactive waste collection system. The 1,514-L (400-gal) stainless steel sump tank is approximately 1.06 m (42 in.) in diameter in the center and extends approximately 2.1 m (7 ft) to the tip of the cone. The top of Tank V-9 is approximately 2.1 m (7 ft) below ground surface (bgs) and is accessible by a 15.2-cm (6-in.) diameter vent pipe that extends to ground surface. A baffle is located in the tank near the inlet ports. Tank V-9 has two subsurface inlet lines that received wastewater from several TAN sources via the TAN-1704 valve pit. One subsurface outlet line discharged overflow from Tank V-9 to Tanks V-1, V-2, and V-3.

##### **G-1.2 1996 Tank V-9 Characterization Results**

The 1996 remedial investigation/feasibility study estimated that approximately 750 to 950 L (200 to 250 gal) of sludge and 265 L (70 gal) of liquid remain in the conical tank. The volume of material located behind the baffle was not known. The total waste material volume inside the tank was estimated at 1,216 L (320 gal) (Blackmore 1998).

In March 1996, Tank V-9 was sampled and 4 L (1.1 gal) of liquid were collected from a location in the tank 3.05 m (10 ft) bgs and an estimated 1.07 m (3.5 ft) above the conical tank bottom. The sample was collected with a peristaltic pump fitted with Teflon tubing and inserted into the tank through a 15.2-cm (6-in.) pipe extending from the ground surface into the top of the tank at the center. The collected liquid, dark brown in color and containing significant amounts of fine particulate matter, was shipped to an onsite laboratory for analysis. The requested analyses for the sample were anions (Cl, NO<sub>3</sub>, NO<sub>2</sub>, PO<sub>4</sub>, SO<sub>4</sub>, F, and Br), total halogens, total organic carbon, total suspended solids, pH, Contract Laboratory Program (CLP) metals (with Sn, B, and Si added to the target analyte list), polychlorinated biphenyls (PCBs), volatile organic compounds, semivolatile organic compounds, gamma spectroscopy, U/Pu isotopes, and other isotopes including Cm, Am, Sr, Np, Ra, and H<sub>3</sub> (Department of Energy Idaho Operations Office [DOE-ID] 1997).

In April and June of 1996, the solids in Tank V-9 were sampled using a long-handled device with a detachable sample bomb. The sampling device was inserted into the tank solids, and the sample was collected by sliding a 10.2-cm (4-in.) length of aluminum housing over the material using a T-handle. Outside the tank, the collected material was transferred from the bomb into a stainless steel pan and then into sample jars (two 250-mL sample containers during the first sampling event) with a stainless steel spoon. The tank solids, which had the consistency of mud and contained a large amount of organic debris such as twigs and straw, were collected from a location 4.1 m (13.5 ft) bgs and an estimated 15.2 cm (6 in.) to 0.3 m (1 ft) above the tank bottom. During the second sampling event performed in the same manner in June of 1996, 1,000 mL of tank solids were collected. The majority of the collected material was submitted to an onsite laboratory for the following analyses (in duplicate): anions (Cl, NO<sub>3</sub>, NO<sub>2</sub>, PO<sub>4</sub>, SO<sub>4</sub>, F, and Br), total halogens, total organic carbon, percent moisture, pH, particle-size distribution, density, CLP metals (with Sn, B, and Si added to the target analyte list), volatile organic compounds, gamma spectroscopy, U/Pu isotopes, and other isotopes including Cm, Am, Sr, Np, and Ra. Additional sample material was stored under refrigeration until it was shipped to a commercial laboratory in October 1996, for analysis of the CLP target compound list that included semivolatile organic compounds and PCBs using SW-846 methods (DOE-ID 1997).

The analytical results for the liquid sample and two sludge samples collected from Tank V-9 in 1996 are presented in the remedial investigation/feasibility study and also in Appendix H of this Remedial Design/Remedial Action Work Plan. The results of the sampling and analysis indicate that the contents of Tank V-9 are similar in chemical nature to those of Tanks V-1, V-2, and V-3. The sample results reported high concentrations of organic compounds (e.g., trichloroethene and PCBs) and radionuclides (e.g., Cs, U, Am, Pu, and H<sub>3</sub>) (DOE-ID 1997). Because of the high concentration of fissile materials in the tank, a criticality evaluation was conducted in 1998. The evaluation was not conclusive in determining if the mass of fissile material in Tank V-9 was sufficient for a criticality event and recommended that additional sampling be conducted (Blackmore 1998).

Based on the recommendation from the 1998 criticality evaluation, nine samples (including one duplicate) were collected from Tank V-9 in April and May 2001, with four of the samples collected from behind the baffle. The remaining sections of this report detail the following aspects of the 2001 sampling effort: sampling equipment and testing, sampling procedures and details, analytical data summary, and lessons learned. Summaries of the raw analytical data and data validation reports are provided in Attachment G-1.

## G-2. SAMPLING EQUIPMENT AND TESTING

The original concept proposed for the Tank V-9 sampling equipment was that of a flexible composite liquid waste sampler (COLIWASA) to be diverted to the backside of the baffle through a cable-articulated pipe section. The first full-scale mock-up investigated the feasibility of double articulation of the sampler guide to prevent the sampling device from scraping the wall of the tank while collecting the samples. Testing indicated that there simply was not enough headspace in the tank for double articulation. Consideration was given to the possibility of collecting a single continuous top-to-bottom sample instead of several discrete samples. This technique would have produced less tank disturbance, but the wide range of possible sludge consistencies might have clogged the sampling tube.

Discrete samples require the use of a valved sampler that could be lowered to a known sampling interval, opened, filled, and remotely closed. A commercially available Discrete Sludge Sampler was purchased and tested in a variety of simulated sludges at the Science Applications International Corporation's STAR Center. The simulated sludge consisted primarily of local clay soil and water in various proportions. The valve mechanism failed to operate properly in many of the thicker test sludges.

Project engineers designed a new sampler with a pointed piston valve mechanism. The new sampler performed well in a wide variety of sludges and liquids. The sampler was made from readily available stainless steel and polyvinyl chloride (PVC) pipe fittings. The body of the sampler was a 7-in. long piece of 2-in. schedule 80 PVC pipe. The pipe was threaded into a modified stainless steel bell reducer, which had a threaded attachment for the handle and a pair of setscrews to lock a 1/2-in. nylon rod that operated the piston at the other end of the pipe (Figure G-1). The sampler would hold a maximum volume of 320 mL; the target sample volume was 200 to 300 mL. Criticality concerns dictated that no more than 5.5 L of material could be removed from the tank during the entire sampling effort. The sampler was attached to a 7-ft flexible hollow handle containing a flexible rod for valve actuation. These, in turn, were connected to shorter sections of a rigid handle and rod to navigate the small-diameter pipe from the tank, up to ground level, and into the glove bag at the samplers' station. An aluminum guide track with a cable-actuated hinge was used to direct the sampler either straight down for the on-axis samples or offset for the off-axis samples. A lamp and tiny video camera were lowered into the tank for initial reconnaissance and continuous monitoring of the sampling activities.

All equipment was built full scale for rigorous testing and operator training at the STAR Center. A platform was built to simulate ground level with the tank mock-up in full view below. During the testing and training phases, minor modifications and fine-tuning improved the equipment. Before mobilization of the equipment to the V-Tank site was authorized, several demonstrations of the equipment were held; the sampling operators were trained in every aspect of setup, operation, and disposal of the sampling equipment, with special emphasis on full containment of the samples and equipment within the glove bag enclosure. Once the final version of the detailed procedure was completed, more training was conducted, video-taped, and timed to document the ability to comply verbatim with each step of the procedure, and to estimate the stay-times that would be required in the radiation field. As low as reasonably achievable (ALARA) practices were emphasized throughout the training.

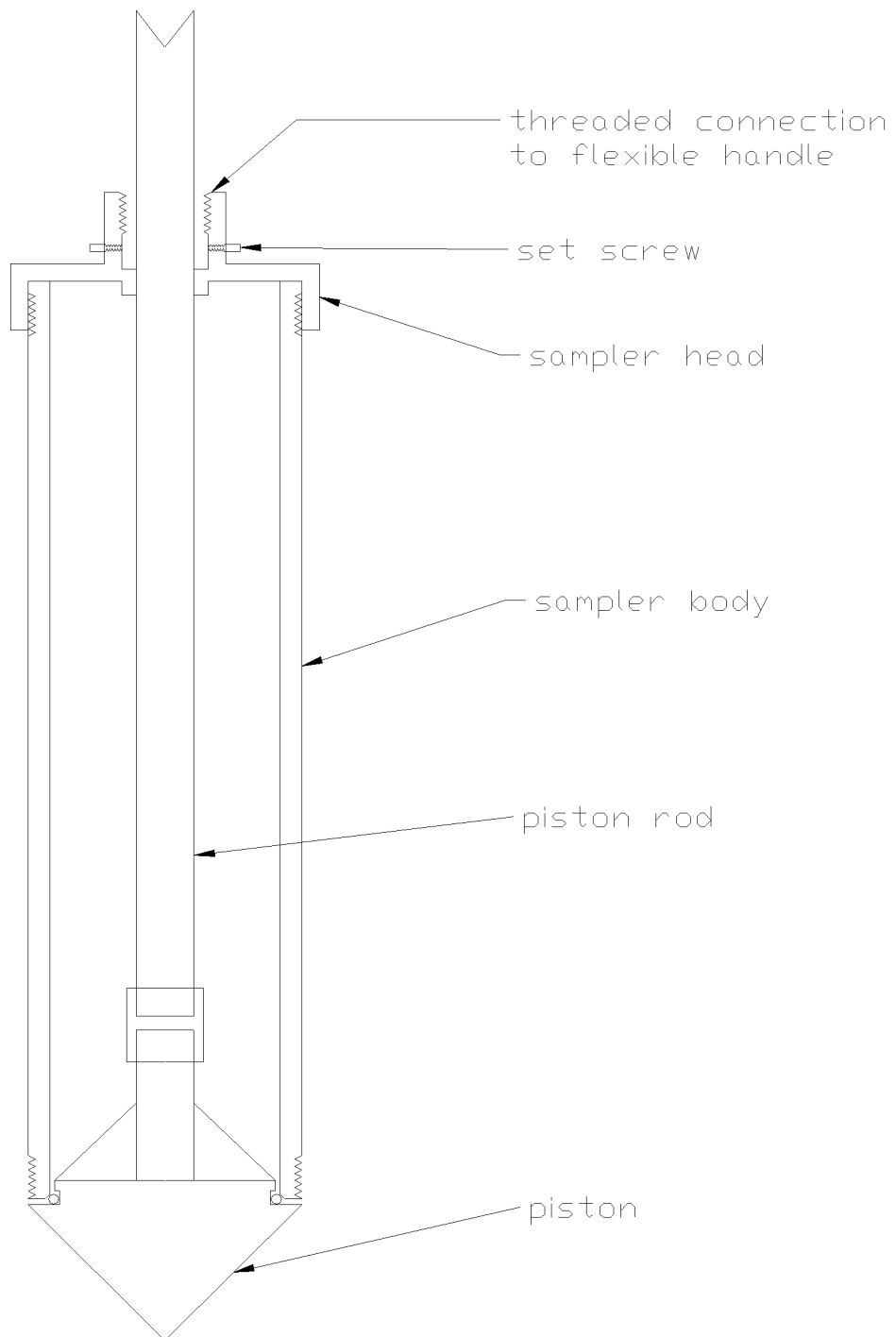


Figure G-1. Sampler Assembly Detail.

## **G-3. TANK V-9 SAMPLING PROCEDURES AND DETAILS**

### **G-3.1 Sampling Procedures**

The sampling procedures specified in the technical procedure “Tank V-9 Sampling Procedure,” (WESTON 2001) were generally segregated into three sections: setup, sampling, and disassembly. Consensus was that the setup and disassembly portions were to be “general intent” procedures that could be less rigorously defined with allowances for operator discretion and leeway in the order of operations. The sampling steps that include any steps that disturbed the tank contents in any way required an extremely detailed description with verbatim compliance and a check-off spot for completion of each step. Because of a possible criticality, no chances could be taken that an inadvertently skipped step in the procedure could cause a problem. The procedure is briefly summarized as follows:

1. Build a tent over the tank access
2. Add on extension pipes from ground level up to a comfortable working height
3. Insert lamp and camera for initial reconnaissance and field-verification of dimensions
4. Insert the aluminum guide channel
5. Assemble the glove bag
6. Collect and recover the samples, passing them out of the glove bag into approved shipping containers
7. Decontaminate the equipment
8. Pass equipment out of the glove bag into waste storage containers
9. Disassemble the glove bag
10. Remove extension pipes
11. Remove tent.

Execution followed the written procedure with very few field modifications:

1. The sampling tent was installed without incident. The 10 by 12-ft tent was built on a wooden platform; its primary function was to protect the samplers and equipment from wind and rain.
2. Samplers installed extension pipes. These pipes raised the tank access from below grade level to waist height and had connections for a high-efficiency particulate air vacuum filter and continuous gas monitoring of the tank.
3. The lamp and camera were installed to verify that initial conditions matched those photographed in 1996. The camera-mounting rod was used to measure the actual depth from the top of the extension pipes to the top of the tank for comparison with the assumed depth. Based upon existing information, the assumed depth to the top of the tank had been estimated to be 10 ft, 9.25 in., and the actual depth was measured at 10 ft, 11.25 in. A hold point in the procedure allowed for overnight re-manufacture of some sampling equipment to match the measured depth.

4. The aluminum guide channel was inserted, and its actuator tested. Initial testing of the articulated section disturbed the surface of the water in the tank, leading to the determination that the level in the tank was at least an inch higher than previously seen in 1996.
5. The glove bag was reassembled in the same orientation with respect to the baffle, as it was for all of the training exercises. The soft-sided glove bag was suspended from a steel and wood frame. Overall dimensions were 24 in. wide, 48 in. long, and 30 in. high (Figure G-2). Directly above the tank access was an 8-ft tall sealed “chimney” that provided containment for the long flexible handle. During sample recovery, the handle could be stored above the “trap door” in the chimney, leaving only the sampler body in the glove bag (Figure G-3). The floor of the glove bag supported a vise that was used to clamp the sampler/handle at any elevation.
6. The samples were collected following verbatim compliance with the steps laid out in the work order package. The general procedure was to lower the sampler into the tank headspace by adding 24-in. handle segments, as needed. The guide channel was then articulated to divert the sampler to the backside of the tank baffle. The sampler was then lowered to the beginning of the sample interval. At this point the piston was extended, opening the sampler. The material was allowed to slump back into the void created by the piston. Finally, the sampler body was advanced to “catch up” to the piston, capturing the sample inside the PVC pipe section. Once the sampler was raised up to the glove bag, threaded PVC caps replaced the pointed piston and the handle attachment. The sample was inspected briefly to confirm that sufficient material had been collected. In one instance (1RD005), the sampler had not been properly sealed and there was virtually no sample. The sampling procedure allowed the operators to make another collection attempt from the same location. In three instances, field changes to the sampling procedure were needed that required multiple INEEL approvals.

The first field change addressed the difficulty encountered in pushing the sampler to Zone 5 (for Sample 1RD006). In the first attempt to collect this sample, the operators encountered layers of crusty sediment behind the baffle. Because this crusty material was substantially different from the practice sludge, the operators decided not to risk damage to the sampling equipment. The sampling procedure was revised to allow them to abandon this sample, collect the remaining samples, and return to Zone 5 at a later date for another attempt to collect 1RD006. After encountering the same crusty layers at the same depths in collecting the two samples from Zone 4, the operators agreed that they could have collected Sample 1RD006 without damaging the sampler.

The second change was incorporated to give the sampling operators some discretion in further attempts to collect Sample 1RD006. The change authorized a second attempt to collect the sample from the exact same location as the first attempt. In the event of another failure, this change allowed third and fourth attempts to collect a sample from slightly different locations within the boundaries of Zone 5. The second attempt in the original location was successful in collecting the sample, and the remaining options were not exercised.

The third change allowed a minor deviation in the recovery of the last sample (1RD006). As the sampler was being withdrawn from the tank, it slipped out of the aluminum guide track and became stuck in the extension pipe. Verbatim compliance prohibited re-alignment, as it would require lowering the sample back into the headspace of the tank. The change recognized that this action would not further disturb the tank contents or compromise the sample itself. The sampler was realigned, and the sample recovered without further incident.



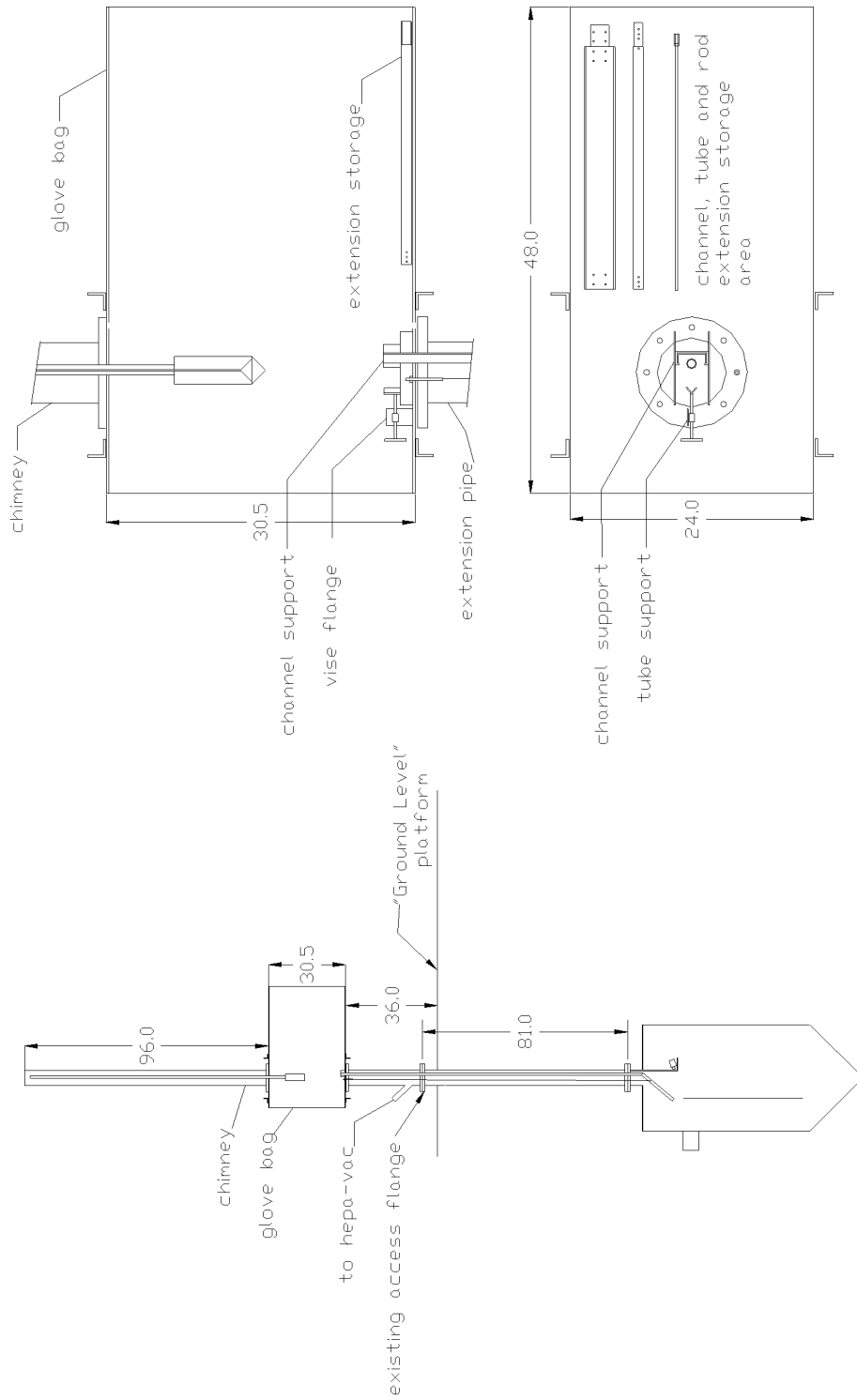


Figure G-2. Sample System and Glove Bag Detail.

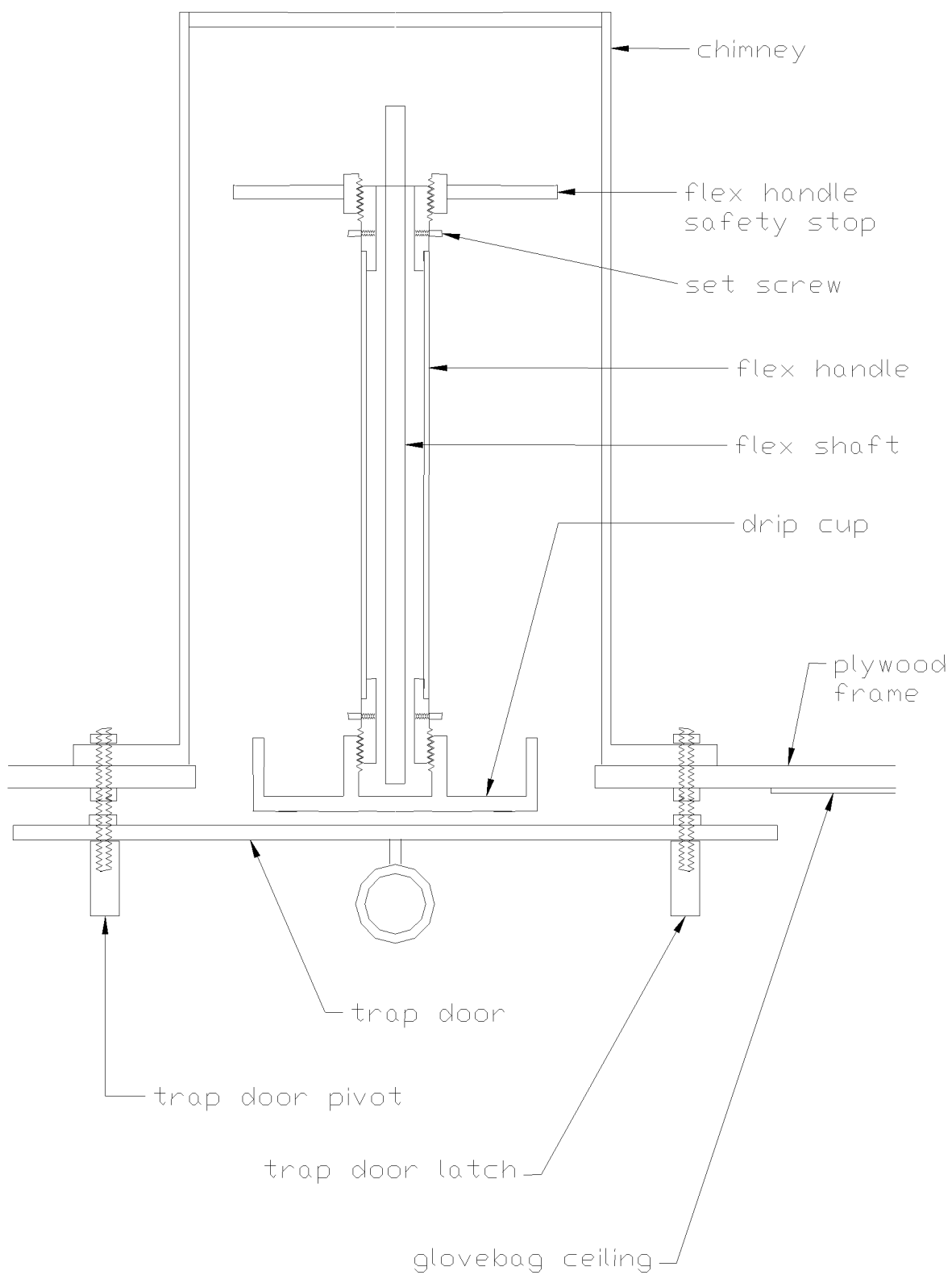


Figure G-3. Chimney Assembly and Contents.

In all cases, the samples were recovered and transferred out of the glove bag. At each recovery, initial measurements were taken to confirm that the radiological limits specified in the radiological work permit were not exceeded. The glove bag was cleaned up and waste material was transferred out before each subsequent sample attempt was initiated.

7. When all samples had been recovered, the equipment was cleaned of gross contamination. The equipment was to be saved for further sampling, if necessary.
8. Most of the equipment had been designed to allow for disassembly within the glove bag without breaking containment. The long, flexible handle was cut into 2-ft lengths. Everything was removed from the glove bag except the two flanges that were too large to fit through the pass-out sleeve.
9. The glove bag was then collapsed around these flanges and packaged in a drum for disposal.
10. The extension pipes were removed and placed in a drum for disposal. One 12-in. section of pipe was left in place to raise the tank access above ground level and was capped with a blind flange.
11. The tent was removed.

### G-3.2 Sample Details

Beginning on April 30, 2001, nine samples (including a quality control duplicate) were collected from eight sample zones (Figure G-4) within Tank V-9. The quality control duplicate was taken from Zone 4. The six off-axis zones are each approximately 18 in. long, while the two on-axis sample zones are each approximately 27 in. long. Table G-1 specifies the sample number, location, depth, and date of collection. Individual sample volumes were intended to be between 200 and 300 mL, with a maximum total sample volume of 2.9 L, well within the specified limit of 5.5 L. The sampling procedure included a check to ensure that the sample container was at least half full (160 mL). Weights or volumes of the samples were not recorded due to the ALARA consideration. Therefore, the sample size is reported as approximately 200 to 300 mL. The sample depths in Table G-1 are measured from the top of the tank to the top of the sampler tube.

Table G-1. Sample Numbers, Locations, Depths, and Collection Dates.

Sample Number	Sample Location	Depth from Top of Tank (in.)	Sample Collection Date
1RD001	Zone 1; on tank center line axis	20	April 30, 2001
1RD002	Zone 2; on tank center line axis	56	April 30, 2001
1RD003	Zone 3; off-axis, behind baffle	20	May 1, 2001
1RD004	Zone 4; off-axis, behind baffle	38	May 2, 2001
1RD005	Zone 4; off-axis, behind baffle (duplicate)	38	May 3, 2001
1RD006	Zone 5; off-axis, behind baffle	56	May 8, 2001
1RD007	Zone 6; off-axis, opposite baffle	20	May 3, 2001
1RD008	Zone 7; off-axis, opposite baffle	38	May 3, 2001
1RD009	Zone 8; off-axis, opposite baffle	56	May 7, 2001

Zones 1 and 2 were shallow and deep, respectively, on the centerline of the tank. These samples were collected first such that materials from the other zones would not drip on the surface of Zone 1 and cross-contaminate the samples. Originally, the procedure called for samples to be taken from Zones 3, 4, and 5, in that order, all from the same planimetric position. A duplicate of the Zone 4 sample would then be collected approximately 5 in. offset from this position by rotating the guide channel 10 degrees. During full-scale testing at the STAR Center, it was noted that the duplicate always had a higher sludge-to-water ratio than the original. This was attributed to the action of removing the Zone 3 sample, which created a water channel down toward the original Zone 4 sample location. The final procedure specified that Zone 5 would be sampled after Zone 3 and also allowed the Zone 4 samples to be collected under identical conditions, each 10 degrees (left and right) from the Zone 3 and 5 sampling locations. The last samples to be collected would then be from Zones 6, 7, and 8. Specific details of the samples are summarized below.

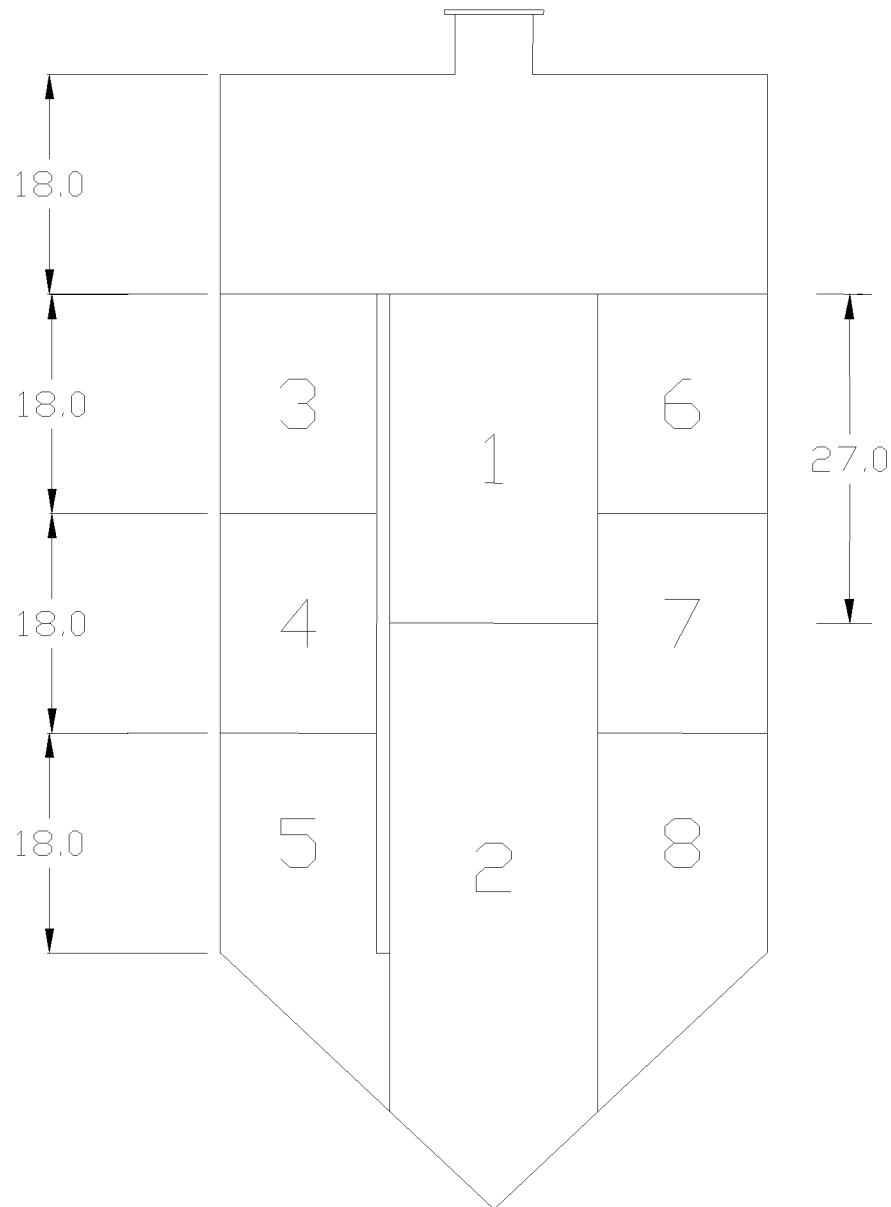


Figure G-4. Tank V-9 Sample Zones.

1. Zone 1 – Sample 1RD001: This sample was collected on the afternoon of April 30, 2001, at 2:00 p.m. The sampler was estimated to be about three-quarters full, and the sample was primarily water. Near contact beta/gamma reading was 3 mR/h.
2. Zone 2 – Sample 1RD002: This sample was also collected on April 30, 2001, at approximately 3:30 p.m. The sampler was inserted to full depth without effort. The sampler was completely full, and the sample had the look and consistency of grease. Near contact beta/gamma reading was 50 mR/h.
3. Zone 3 – Sample 1RD003: This sample was collected on the morning of May 1, 2001, at 11:00 a.m. The sampler was reported to be about one-half full, and the sample was primarily water. Near contact beta/gamma reading was 10 mR/h.
4. Zone 5 – Sample 1RD006: This sample was first attempted on the morning of May 1, 2001. The operators reported significant resistance while attempting to reach Zone 5. Several distinct crusty layers were encountered in Zones 4 and 5. Because the sampling team did not want to risk damaging the sampling apparatus at this early stage of the sampling, the Zone 5 sample was abandoned. The procedure was changed to allow the sampling team to collect the remaining samples before returning to Zone 5.
5. Zone 4 – Sample 1RD004: This sample was collected on the afternoon of May 2, 2001, at about 2:00 p.m. The sampler was full to within 1 in. of the top. The sample, which looked like a purple dye, contained sandy material in the threads of the sample body. Near contact beta/gamma reading was 103 mR/h. The operators reported the same layered crusty conditions noted for the attempt in Zone 5.
6. Zone 4 – Sample 1RD005: This sample was first attempted on the afternoon of May 2, 2001. The sample was captured inside the sampler body, but was lost when the piston lost its seal. During retrieval, the wrong setscrew was inadvertently loosened. The procedure steps allowing a second attempt for “insufficient sample” were followed. The second attempt at about 11:00 a.m. on May 3, 2001, was successful. The sampler was full to within 3/8 in. of the top, and the sample consisted of a mixture of sludge and a gray liquid. Near contact beta/gamma reading was 80 mR/h. The operators noted the same crusty layers as found in the first attempt at Zone 4.
7. Zone 6 – Sample 1RD007: This sample was collected at about 12:00 p.m. on May 3, 2001. The sampler was full to within 3/8 in. of the top with liquid and sludge. The sample appeared to have some grass mixed in with it. Near contact beta/gamma reading was 9 mR/h.
8. Zone 7 – Sample 1RD008: This sample, collected at about 4:00 p.m. on the afternoon of May 3, 2001, was filled to within 1/2 in. of the top. There was no appearance of crusty layers in this zone. Near contact beta/gamma reading was 100 mR/h.
9. Zone 8 – Sample 1RD009: This sample, collected May 7, 2001, at around 10:30 a.m., filled the sampler to within 1/8 in. of the top. The sample looked like black grease and contained what appeared to be part of an old rubber glove. Near contact beta/gamma reading was 80 mR/h.
10. Zone 5 – Sample 1RD006 (second attempt): This sample was collected on May 7, 2001, from the same location as the original attempt on May 1. A little extra force was used to push through the last crusty layer. During the retrieval process, the sampler slipped out of the track and wedged in the pipe riser. The procedure was changed to allow for realignment, and the sample was finally recovered on the morning of May 8, 2001, at about 9:00 a.m. The sample looked like grease and included what seemed to be part of a root or stick. Near contact beta/gamma reading was 80 mR/h.

## **G-4. TANK V-9 ANALYTICAL DATA SUMMARY**

The nine samples were delivered to the BWXT Services, Inc., Nuclear Environmental Laboratory Services located in Lynchburg, Virginia. The samples were received at the laboratory from May 2 through May 9, 2001. On May 10, 2001, each sample was placed in a sample tray and homogenized to the extent practical. From each of the nine homogenized samples, five subsamples were collected for isotopic uranium analysis (using alpha spectrometry), as well as percent moisture and bulk density determinations. Additionally, two composites were prepared from the nine homogenized samples by combining approximately 10-mL aliquots from the Zone 1, 3, and 6 samples with 10- to 25-g aliquots from the remaining six samples (BWXT Services 2001a). The composites were analyzed for TCLP metals.

The initial analytical results for the 45 subsamples are presented in Table G-2, and the TCLP metal analytical results for the composites are presented in Table G-3. In addition to the individual data values for the subsamples, Table G-2 also provides maximum, average, standard deviation, and relative percent difference (RPD) values for each group of five subsamples. Specific results are discussed in the following sections. Summaries of the raw data and data validation reports are included in Attachment G-1. Radioanalytical and TCLP metal data validations were performed by Portage Environmental, Inc., in Butte, Montana, and bulk density and moisture content data validations were performed by the INEEL Sample Management Office.

In August 2001, the BWXT Services, Inc., laboratory was requested to perform additional isotopic uranium analyses on the remaining 45 subsample volumes that had been retained by the laboratory. One of the five subsamples from each sampling zone was randomly selected, and an aliquot was collected and split. One of the splits was prepared using acid dissolution, and the other split was prepared using pyrosulfate fusion. Both splits were then analyzed using alpha spectrometry. A duplicate analysis of the Zone 2 fusion split was also performed. Section G-4.4 and Table G-4 present the additional radioanalytical results that were obtained (BWXT Services 2001b), and summaries of the raw data and data validation reports are included in Attachment G-1. The analytical method data validation was performed by Environmental Data Services, Inc., which is located in Indianola, Pennsylvania.

### **G-4.1 Bulk Density and Moisture Content Results**

The bulk density and moisture content analyses were conducted using American Society for Testing and Materials Standards D5057-90 and E949-88, respectively, and the results are presented in Table G-2. For these analyses, the only applicable quality control parameter is the sample duplicate. Since the laboratory essentially performed duplicates (i.e., the five subsamples collected from each original sample) for every sample, data validation evaluated the sample results based on their subsample's RPDs to each other. For each sample, the maximum and minimum values reported for the five subsamples were used for this calculation as follows:

$$RPD = \frac{Max - Min}{Max + Min} \times 200$$

If the RPD was greater than 35% but less than or equal to 50%, the associated sample results were qualified with a "J" validation flag. If the RPD was greater than 50%, the associated sample results were qualified with an "R" validation flag.

Table G-2. Tank V-9 Physical and Radiochemical Analytical Results

Sample Zone	Sample	Subsample Number	Moisture Content (%)	Bulk		U-234			U-235			U-238		
				Density (g/mL)	DVF	Results (pCi/g)	Uncertainty (pCi/g)	Results (pCi/g)	DVF	Results (pCi/g)	Uncertainty (pCi/g)	DVF	Results (pCi/g)	Uncertainty (pCi/g)
1	IRD001	IRD001013A	98.0	1.0	—	1.90E+02	1.58E+01	9.94E+00	j <sup>2</sup>	9.94E+00	1.36E+00	—	1.53E+00	4.62E-01
1	IRD001	IRD001023A	98.3	1.0	—	1.73E+02	1.44E+01	8.98E+00	j <sup>2</sup>	8.98E+00	1.25E+00	—	1.39E+00	4.29E-01
1	IRD001	IRD001033A	97.9	1.0	—	1.91E+02	1.60E+01	1.01E+01	j <sup>2</sup>	1.01E+01	1.42E+00	—	1.69E+00	5.11E-01
1	IRD001	IRD001043A	98.0	1.0	—	1.69E+02	1.41E+01	9.49E+00	j <sup>2</sup>	9.49E+00	1.31E+00	j <sup>3</sup>	7.14E-01	3.15E-01
1	IRD001	IRD001053A	98.1	1.0	—	1.65E+02	1.36E+01	9.94E+00	j <sup>2</sup>	9.94E+00	1.25E+00	—	1.28E+00	3.73E-01
		Maximum	98.3	1.0	—	1.91E+02	1.60E+01	1.01E+01	—	1.01E+01	1.42E+00	—	1.69E+00	5.11E-01
		Average	98.1	1.0	—	1.78E+02	1.48E+01	9.69E+00	—	9.69E+00	1.32E+00	—	1.32E+00	4.18E-01
		Std. Dev.	0.2	0.0	—	1.21E+01	1.06E+00	4.57E-01	—	4.57E-01	7.33E-02	—	3.72E-01	7.64E-02
		RPD (%)	0.4	0.0	—	—	—	—	—	—	—	—	—	—
2	IRD002	IRD002013A	46.9	0.8	R	1.55E+04	1.25E+03	6.05E+02	j <sup>2</sup>	6.05E+02	6.60E+01	—	1.33E+02	2.39E+01
2	IRD002	IRD002023A	46.7	2.7	R	5.33E+03	4.46E+02	2.31E+02	j <sup>2</sup>	2.31E+02	3.45E+01	—	1.14E+02	2.26E+01
2	IRD002	IRD002033A	46.1	1.5	R	6.93E+03	5.70E+02	2.55E+02	j <sup>2</sup>	2.55E+02	3.49E+01	—	1.11E+02	2.08E+01
2	IRD002	IRD002043A	46.3	1.4	R	1.12E+04	9.08E+02	3.34E+02	j <sup>2</sup>	3.34E+02	4.26E+01	—	1.36E+02	2.40E+01
2	IRD002	IRD002053A	47.6	3.4	R	8.85E+03	7.30E+02	3.85E+02	j <sup>2</sup>	3.85E+02	4.78E+01	—	1.00E+02	2.05E+01
		Maximum	47.6	3.4	—	1.55E+04	1.25E+03	6.05E+02	—	6.05E+02	6.60E+01	—	1.36E+02	2.40E+01
		Average	46.7	2.0	—	9.56E+03	7.81E+02	3.62E+02	—	3.62E+02	4.52E+01	—	1.19E+02	2.24E+01
		Std. Dev.	0.6	1.1	—	3.98E+03	3.14E+02	1.49E+02	—	1.49E+02	1.29E+01	—	1.53E+01	1.66E+00
		RPD (%)	3.2	123.8	—	—	—	—	—	—	—	—	—	—
3	IRD003	IRD003013A	97.4	1.0	—	8.42E+01	6.94E+00	4.49E+00	j <sup>2</sup>	4.49E+00	5.52E-01	—	6.69E-01	1.81E-01
3	IRD003	IRD003023A	97.1	1.0	—	8.96E+01	7.34E+00	5.22E+00	j <sup>2</sup>	5.22E+00	6.09E-01	—	8.31E-01	1.93E-01
3	IRD003	IRD003033A	93.2	1.0	—	8.57E+01	7.02E+00	5.66E+00	j <sup>2</sup>	5.66E+00	6.33E-01	—	8.83E-01	1.92E-01
3	IRD003	IRD003043A	100.4	1.0	—	8.62E+01	7.04E+00	4.83E+00	j <sup>2</sup>	4.83E+00	5.71E-01	—	5.91E-01	1.58E-01
3	IRD003	IRD003053A	97.0	1.0	—	8.26E+01	6.80E+00	4.21E+00	j <sup>2</sup>	4.21E+00	5.14E-01	—	6.24E-01	1.60E-01
		Maximum	100.4	1.0	—	8.96E+01	7.34E+00	5.66E+00	—	5.66E+00	6.33E-01	—	8.83E-01	1.93E-01
		Average	97.0	1.0	—	8.57E+01	7.03E+00	4.88E+00	—	4.88E+00	5.76E-01	—	7.20E-01	1.77E-01
		Std. Dev.	2.6	0.0	—	2.61E+00	1.98E-01	5.76E-01	—	5.76E-01	4.69E-02	—	1.30E-01	1.69E-02
		RPD (%)	7.4	0.0	—	—	—	—	—	—	—	—	—	—
4	IRD004	IRD004013A	52.5	1.7	j <sup>1</sup>	1.38E+04	1.12E+03	7.19E+02	—	7.19E+02	7.42E+01	—	3.66E+02	4.47E+01
4	IRD004	IRD004023A	53.1	1.4	j <sup>1</sup>	2.22E+04	1.78E+03	8.98E+02	—	8.98E+02	8.91E+01	—	3.08E+02	3.98E+01
4	IRD004	IRD004033A	43.7	1.6	j <sup>1</sup>	7.44E+03	6.22E+02	3.54E+02	—	3.54E+02	4.78E+01	—	3.03E+02	4.31E+01

Table G-2. (continued).

Sample Zone	Sample	Subsample Number	Moisture Content (%)	Bulk Density (g/mL)	DVF	U-234 Results (pCi/g)	U-234 Uncertainty (pCi/g)	U-235 Results (pCi/g)	U-235 DVF	U-235 Uncertainty (pCi/g)	U-238 Results (pCi/g)	DVF	U-238 Uncertainty (pCi/g)
4	IRD004	IRD004043A	54.8	1.8	J <sup>1</sup>	1.86E+04	1.51E+03	7.57E+02	—	8.29E+01	3.78E+02	—	5.04E+01
4	IRD004	IRD004053A	39.5	2.2	J <sup>1</sup>	8.41E+03	6.92E+02	3.74E+02	—	4.75E+01	2.38E+02	—	3.51E+01
		Maximum	54.8	2.2	—	2.22E+04	1.78E+03	8.98E+02	—	8.91E+01	3.78E+02	—	5.04E+01
		Average	48.7	1.7	—	1.41E+04	1.14E+03	6.20E+02	—	6.83E+01	3.19E+02	—	4.26E+01
		Std. Dev.	6.7	0.3	—	6.38E+03	5.04E+02	2.43E+02	—	1.96E+01	5.62E+01	—	5.69E+00
		RPD (%)	32.4	44.4	—	—	—	—	—	—	—	—	—
4	IRD005	IRD005013A	55.7	2.4	R	7.03E+03	5.82E+02	4.00E+02	—	4.96E+01	4.14E+02	—	5.08E+01
4	IRD005	IRD005023A	58.5	1.3	R	2.16E+04	1.74E+03	1.22E+03	—	1.16E+02	1.05E+03	—	1.03E+02
4	IRD005	IRD005033A	64.7	1.5	R	8.21E+03	6.74E+02	4.78E+02	—	5.30E+01	4.28E+02	—	4.88E+01
4	IRD005	IRD005043A	55.9	1.5	R	1.32E+04	1.06E+03	7.73E+02	—	7.77E+01	5.61E+02	—	6.03E+01
4	IRD005	IRD005053A	55.2	1.9	R	2.01E+04	1.61E+03	1.14E+03	—	1.08E+02	1.33E+03	—	1.23E+02
		Maximum	64.7	2.4	—	2.16E+04	1.74E+03	1.22E+03	—	1.16E+02	1.33E+03	—	1.23E+02
		Average	58.0	1.7	—	1.40E+04	1.13E+03	8.02E+02	—	8.09E+01	7.57E+02	—	7.72E+01
		Std. Dev.	4.0	0.4	—	6.67E+03	5.28E+02	3.73E+02	—	3.06E+01	4.12E+02	—	3.37E+01
		RPD (%)	15.8	59.5	—	—	—	—	—	—	—	—	—
5	IRD006	IRD006013A	43.6	1.5	—	2.38E+04	1.99E+03	1.13E+03	—	1.34E+02	8.18E+02	—	1.06E+02
5	IRD006	IRD006023A	39.6	1.6	—	2.75E+04	2.30E+03	1.25E+03	—	1.46E+02	9.72E+02	—	1.22E+02
5	IRD006	IRD006033A	37.0	1.8	—	1.73E+04	1.46E+03	1.11E+03	—	1.31E+02	1.19E+03	—	1.37E+02
5	IRD006	IRD006043A	40.8	1.6	—	1.46E+04	1.23E+03	7.47E+02	—	9.57E+01	9.30E+02	—	1.12E+02
5	IRD006	IRD006053A	41.0	1.9	—	1.37E+04	1.17E+03	8.21E+02	—	1.06E+02	7.41E+02	—	9.87E+01
		Maximum	43.6	1.9	—	2.75E+04	2.30E+03	1.25E+03	—	1.46E+02	1.19E+03	—	1.37E+02
		Average	40.4	1.7	—	1.94E+04	1.63E+03	1.01E+03	—	1.23E+02	9.30E+02	—	1.15E+02
		Std. Dev.	2.4	0.2	—	6.02E+03	4.95E+02	2.16E+02	—	2.09E+01	1.71E+02	—	1.49E+01
		RPD (%)	16.4	23.5	—	—	—	—	—	—	—	—	—
6	IRD007	IRD007013A	94.0	1.0	—	5.04E+02	4.14E+01	2.65E+01	—	3.04E+00	1.07E+01	—	1.63E+00
6	IRD007	IRD007023A	94.3	1.1	—	4.80E+02	3.91E+01	2.83E+01	—	3.06E+00	1.01E+01	—	1.48E+00
6	IRD007	IRD007033A	93.9	1.0	—	3.86E+02	3.16E+01	1.88E+01	—	2.31E+00	5.59E+00	—	1.07E+00
6	IRD007	IRD007043A	94.4	1.1	—	5.08E+02	4.16E+01	2.79E+01	—	3.16E+00	1.03E+01	—	1.60E+00
6	IRD007	IRD007053A	94.3	1.0	—	4.48E+02	3.68E+01	2.39E+01	—	2.84E+00	7.82E+00	—	1.37E+00
		Maximum	94.4	1.1	—	5.08E+02	4.16E+01	2.83E+01	—	3.16E+00	1.07E+01	—	1.63E+00
		Average	94.2	1.0	—	4.65E+02	3.81E+01	2.51E+01	—	2.88E+00	8.90E+00	—	1.43E+00
		Std. Dev.	0.2	0.1	—	5.03E+01	4.13E+00	3.91E+00	—	3.40E+01	2.17E+00	—	2.26E+01



Table G-2. (continued).

Sample Zone	Sample	Subsample Number	Moisture Content (%)	Bulk Density (g/mL)	U-234 Results (pCi/g)	DVF	U-234 Uncertainty (pCi/g)	U-235 Results (pCi/g)	DVF	U-235 Uncertainty (pCi/g)	U-238 Results (pCi/g)	DVF	U-238 Uncertainty (pCi/g)
		RPD (%)	0.5	9.5	—	—	—	—	—	—	—	—	—
7	IRD008	IRD008013A	66.9	1.4	—	—	3.35E+02	1.73E+02	—	2.16E+01	2.50E+01	—	6.76E+00
7	IRD008	IRD008023A	67.5	1.3	—	—	3.30E+02	1.69E+02	—	2.07E+01	1.85E+01	—	5.58E+00
7	IRD008	IRD008033A	66.4	1.3	—	—	3.67E+02	1.41E+02	—	1.96E+01	1.64E+01	J <sup>3</sup>	5.84E+00
7	IRD008	IRD008043A	66.5	1.2	—	—	3.06E+02	1.65E+02	—	2.16E+01	1.87E+01	J <sup>3</sup>	6.55E+00
7	IRD008	IRD008053A	67.1	1.3	—	—	3.78E+02	1.90E+02	—	2.30E+01	2.95E+01	—	7.34E+00
		Maximum	67.5	1.4	—	—	3.78E+02	1.90E+02	—	2.30E+01	2.95E+01	—	7.34E+00
		Average	66.9	1.3	—	—	3.43E+02	1.68E+02	—	2.13E+01	2.16E+01	—	6.41E+00
		Std. Dev.	0.4	0.1	—	—	2.92E+01	1.77E+01	—	1.26E+00	5.45E+00	—	7.11E-01
		RPD (%)	1.6	15.4	—	—	—	—	—	—	—	—	—
8	IRD009	IRD009013A	62.0	1.6	R	R	2.29E+02	1.07E+02	—	1.57E+01	1.22E+01	J <sup>3</sup>	4.77E+00
8	IRD009	IRD009023A	62.4	2.3	R	R	2.75E+03	1.34E+02	—	1.84E+01	1.11E+01	J <sup>3</sup>	4.60E+00
8	IRD009	IRD009033A	61.6	1.1	R	R	3.27E+02	1.39E+02	—	1.83E+01	1.48E+01	J <sup>3</sup>	5.08E+00
8	IRD009	IRD009043A	59.9	1.7	R	R	2.74E+02	9.89E+01	—	1.37E+01	1.38E+01	—	4.43E+00
8	IRD009	IRD009053A	63.4	1.2	R	R	3.04E+02	1.26E+02	—	1.69E+01	1.14E+01	J <sup>3</sup>	4.40E+00
		Maximum	63.4	2.3	—	—	3.27E+02	1.39E+02	—	1.84E+01	1.48E+01	—	5.08E+00
		Average	61.9	1.6	—	—	2.82E+02	1.21E+02	—	1.66E+01	1.27E+01	—	4.66E+00
		Std. Dev.	1.1	0.5	—	—	4.01E+01	1.98E+01	—	2.26E+00	1.65E+00	—	2.77E-01
		RPD (%)	5.7	70.6	—	—	—	—	—	—	—	—	—

RPD = relative percent difference using maximum and minimum values among the five subsamples from each zone.  
DVF = data validation flag. Data validation flags apply to results in the preceding column. If a data validation flag is not shown, the results in the preceding column are unqualified and can be categorized as definitive data with no associated quality control deficiencies.

R = The accuracy of the data is so questionable that it is recommended that the data not be used. For the three bulk density samples 1RD002, 1RD005, and 1RD009, an "R" flag is assigned because the RPDs between the subsamples (using the highest and lowest subsample values) were greater than 50% (i.e., 123.8%, 59.4%, and 70.6%, respectively).

J = estimated value

J<sup>1</sup> = The data are detectable at the reported values, but the reported values are only estimates due to anomalies in the quality control data. For the bulk density sample 1RD004, the "J<sup>1</sup>" flag is assigned because the RPD between the subsamples (using the highest and lowest subsample values) was greater than 35% but less than or equal to 50% (i.e., 44.4%).

J<sup>2</sup> = The analysis was performed and radioactivity was detected. However, the result is questionable due to analytical and/or laboratory quality control anomalies and should therefore be used only as an estimated quantity. For the U-235 analyses for samples 1RD001, 1RD002, and 1RD003, one of the three laboratory control sample recovery results (137.7%) was greater than the prescribed limits of 70–130%, which may indicate high biased sample results.

J<sup>3</sup> = The analysis was performed and radioactivity was detected. However, the result is questionable due to analytical and/or laboratory quality control anomalies and should therefore be used only as an estimated quantity. For the seven U-238 subsample analyses assigned the "J<sup>3</sup>" flag, the results are statistically positive, but the activities are between 2 and 3 times the associated uncertainty instead of greater than 3 times the uncertainty for unflagged values.

g/mL = grams per milliliter  
pCi/g = picocuries per gram

Table G-3. Tank V-9 TCLP Analytical Results.

Sample Number	Analyte	Analytical Method	Results (µg/L)	Data Validation Flag	MDL (µg/L)	Required Detection Limit (µg/L)	Regulatory Level (µg/L)	Exceeds Regulatory Limit
1RD01001TI	Arsenic	ICP--6010A	37.8	U	37.8	250	5,000	NO
1RD01001TI	Barium	ICP--6010A	921	J <sup>1</sup>	4.44	1,000	100,000	NO
1RD01001TI	Cadmium	ICP--6010A	970	J <sup>2</sup>	4.44	50	1,000	NO
1RD01001TI	Chromium	ICP--6010A	234	—	17.8	250	5,000	NO
1RD01001TI	Lead	ICP--6010A	84.4	U	84.4	250	5,000	NO
1RD01001TI	Mercury	CV--7470A	133	—	4	2	200	NO
1RD01001TI	Selenium	ICP--6010A	48.9	U	48.9	50	1,000	NO
1RD01001TI	Silver	ICP--6010A	15.6	U, UJ	15.6	250	5,000	NO
1RD01002TI	Arsenic	ICP--6010A	46.2	B, U <sup>1</sup>	37.8	250	5,000	NO
1RD01002TI	Barium	ICP--6010A	969	J <sup>1</sup>	4.44	1,000	100,000	NO
1RD01002TI	Cadmium	ICP--6010A	1,000	J <sup>2</sup>	4.44	50	1,000	YES
1RD01002TI	Chromium	ICP--6010A	276	—	17.8	250	5,000	NO
1RD01002TI	Lead	ICP--6010A	89.8	B	84.4	250	5,000	NO
1RD01002TI	Mercury	CV--7470A	226	—	4	2	200	YES
1RD01002TI	Selenium	ICP--6010A	64	B, U <sup>1</sup>	48.9	50	1,000	NO
1RD01002TI	Silver	ICP--6010A	15.6	U, UJ	15.6	250	5,000	NO

Method Legend:

MDL = method detection limit

ICP = inductively coupled plasma atomic emission spectrometry

CV = cold vapor atomic absorption spectrometry

Laboratory Concentration Comment (U and B codes) and Data Validation Flag Legend:

B = Analyte concentration greater than the method detection limit (MDL), but less than 10 times the MDL

J = estimated value

J<sup>1</sup> = All barium results have been qualified with a "J<sup>1</sup>" validation flag to denote that the data are detectable at the reported values but that the reported values are only estimates due to low recoveries for continuing calibration verification (89.4% vs. 90–110% recovery criteria), matrix spike (78.5% vs. 80–120% recovery criteria), and matrix spike duplicate (79.0% vs. 80–120% recovery criteria).

J<sup>2</sup> = All cadmium results have been qualified with a "J<sup>2</sup>" validation flag to denote that the data are detectable at the reported values but that the reported values are only estimates due to low recoveries for matrix spike (77.8% vs. 80–120% recovery criteria), matrix spike duplicate (78.6% vs. 80–120% recovery criteria), and analytical spike (-94.8% vs. 75–125% recovery criteria).

U = analyte concentration less than the MDL

U<sup>1</sup> = The arsenic and selenium results for sample 1RD01002TI have been qualified with a "U<sup>1</sup>" validation flag to denote that the sample results are greater than the method detection limits but less than five times the amount of analyte found in the preparation blanks.

UJ = All silver sample results have been qualified with a "UJ" validation flag to denote that the data are non-detectable at the reported values and that the reported values are only estimates due to low recoveries for matrix spike (66.4% vs. 80–120% recovery criteria), matrix spike duplicate (73.4% vs. 80–120% recovery criteria), and analytical spike (64.0% vs. 75–125% recovery criteria).

µg/L = micrograms per liter

Table G-4. Tank V-9 Additional Isotopic Uranium Analyses.

Sample Zone	Sample Number	Sample Size (grams)	Preparation Method	U-234 Results (pCi/g)	DVF	U-234 Uncert. (pCi/g)	U-235 Results (pCi/g)	DVF	U-235 Uncert. (pCi/g)	U-238 Results (pCi/g)	DVF	U-238 Uncert. (pCi/g)
1	IRD05001R9	1.0714	Dissolution	1.36E+02	J	1.12E+01	6.88E+00	J	8.70E-01	7.52E-01	J	2.53E-01
1	IRD05002R9	1.0573	Fusion	1.31E+02	J	1.06E+01	6.78E+00	J	7.98E-01	9.65E-01	J	2.40E-01
			RPD (%)	3.7	—	—	1.5	—	—	24.8	—	—
2	IRD05101R9	0.0103	Dissolution	1.01E+04	J	9.02E+02	4.09E+02	J	6.54E+01	1.60E+02	J	3.76E+01
2	IRD05102R9	0.0106	Fusion	7.87E+03	J	6.76E+02	3.22E+02	J	5.33E+01	1.48E+02	J	3.37E+01
			RPD (%)	24.8	—	—	23.8	—	—	7.8	—	—
2	IRD05102R9	0.0106	Fusion	7.87E+03	J	6.76E+02	3.22E+02	J	5.33E+01	1.48E+02	J	3.37E+01
2-Dup.	0107074-04D	0.01	Fusion	8.46E+03	J	7.33E+02	2.65E+02	J	4.87E+01	3.44E+02	J	5.71E+01
			RPD (%)	7.2	—	—	19.4	—	—	79.7	—	—
3	IRD05201R9	1.1919	Dissolution	7.08E+01	J	6.06E+00	3.85E+00	J	5.27E-01	5.27E-01	J	1.66E-01
3	IRD05202R9	1.0340	Fusion	7.48E+01	J	6.20E+00	4.95E+00	J	6.41E-01	9.49E-01	J	2.39E-01
			RPD (%)	5.5	—	—	25.0	—	—	57.2	—	—
4	IRD05301R9	0.0110	Dissolution	3.08E+04	J	2.54E+03	1.19E+03	J	1.33E+02	3.74E+02	J	5.94E+01
4	IRD05302R9	0.0111	Fusion	3.01E+04	J	2.57E+03	1.15E+03	J	1.31E+02	3.18E+02	J	5.40E+01
			RPD (%)	2.3	—	—	3.4	—	—	16.2	—	—
4	IRD05401R9	0.0265	Dissolution	2.40E+04	J	1.94E+03	1.30E+03	J	1.43E+02	8.45E+02	J	1.04E+02
4	IRD05402R9	0.0258	Fusion	2.33E+04	J	1.88E+03	1.08E+03	J	1.17E+02	7.95E+02	J	9.36E+01
			RPD (%)	3.0	—	—	18.5	—	—	6.1	—	—
5	IRD05501R9	0.0113	Dissolution	5.29E+04	J	4.34E+03	2.35E+03	J	2.39E+02	9.61E+02	J	1.21E+02
5	IRD05502R9	0.0113	Fusion	5.65E+04	J	4.70E+03	2.52E+03	J	2.73E+02	1.06E+03	J	1.45E+02
			RPD (%)	6.6	—	—	7.0	—	—	9.8	—	—
6	IRD05601R9	1.2042	Dissolution	5.67E+02	J	4.56E+01	2.80E+01	J	2.86E+00	1.31E+01	J	1.62E+00
6	IRD05602R9	1.2973	Fusion	5.61E+02	J	4.49E+01	3.09E+01	J	3.05E+00	1.33E+01	J	1.60E+00
			RPD (%)	1.1	—	—	9.8	—	—	1.5	—	—
7	IRD05701R9	0.0258	Dissolution	4.50E+03	J	3.91E+02	1.84E+02	J	2.89E+01	3.06E+01	J	1.06E+01
7	IRD05702R9	0.0283	Fusion	3.93E+03	J	3.26E+02	1.56E+02	J	2.35E+01	2.75E+01	J	8.87E+00

Table G-4. (continued).

Sample Zone	Sample Number	Sample Size (grams)	Preparation Method	U-234 Results (pCi/g)	DVF	U-234 Uncert. (pCi/g)	U-235 Results (pCi/g)	DVF	U-235 Uncert. (pCi/g)	U-238 Results (pCi/g)	DVF	U-238 Uncert. (pCi/g)
8	IRD05801R9	0.0263	RPD (%) Dissolution	13.5 3.43E+03	— J	— 2.91E+02	16.5 1.11E+02	— J	— 2.03E+01	10.7 7.34E+00	— U	— 5.36E+00
8	IRD05802R9	0.0256	Fusion	3.49E+03	J	3.02E+02	1.19E+02	J	2.04E+01	1.05E+01	U	5.79E+00
			RPD (%)	1.7	—	—	7.0	—	—	35.4	—	—
Preparation Method Legend: Dissolution—strong mineral acid dissolution; Fusion—molten salt fusion.												
DVF = Data validation flag. Data validation flags apply to results in the preceding column.												
J = estimated value. The analysis was performed and radioactivity was detected (i.e., the radioanalytical result is statistically positive at the 95% confidence level and is above the minimum detectable activity). However, the result is questionable due to analytical and/or laboratory quality control anomalies (i.e., uranium isotope laboratory control sample recoveries outside acceptable tolerances) and should, therefore, be used only as an estimated (approximated) quantity.												
NOTE: The radionuclide is considered to be present in the sample, but the result may not be an accurate representation of the amount of activity actually present in the sample.												
U = undetected. The analysis was performed, but no radioactivity was detected (i.e., the radioanalytical result was not statistically positive at the 95% confidence level and/or the result was below its minimum detectable activity). The “U” qualifier flag is also applicable to any result reported as zero (0) (+/- an associated uncertainty).												
NOTE: The radionuclide is not considered to be present in the sample.												
RPD = relative percent difference												
pCi/g = picocuries/gram												
Uncert. = Uncertainty												
Dup. = Duplicate												

As discussed in Section G-3.2, the samples collected from Zones 1, 3, and 6 (the uppermost zones in the tank) were primarily liquid, and the samples from the remaining zones exhibited more of a sludge/paste consistency. These observations are confirmed by the analytical data where the average bulk density for the upper zones is 1.0 g/mL and average moisture content ranges from 94.4% in Zone 6 to 98.1% in Zone 1. RPDs for bulk density and moisture content vary from 0.0 to 9.5% and 0.4 to 7.4%, respectively.

The bulk density and moisture content results for the remaining sample zones (2, 4, 5, 7, and 8) vary considerably more. Average bulk density ranges from 1.3 to 2.0 g/mL, and average moisture content ranges from 40.4 to 66.9%. RPDs for bulk density and moisture content data range from 15.4 to 123.8% and from 1.6 to 32.4%. Based upon the RPD criteria above, the bulk density results for Samples 1RD002, 1RD005, and 1RD009 are assigned an “R” validation flag, and the bulk density for 1RD004 is assigned a “J” validation flag.

## **G-4.2 Isotopic Uranium Activity Results**

The 45 subsamples from Tank V-9 were analyzed for U-234, U-235, and U-238 isotopic activity using alpha spectrometry; the results are given in Table G-2, along with the associated uncertainty in the activity measurement and the maximum, average, and standard deviations for the sample group (i.e., the five subsamples). Individual sample sizes, in grams, are provided in the results table in Attachment G-1 of this report. The samples from the upper zones in the tank (Zones 1, 3, and 6) that were primarily liquid (1RD001, 1RD003, and 1RD007) typically display lower uranium activities than the samples collected from the bottom zones that had more of a sludge-like appearance. For the three upper samples, uranium activity is lowest in the sample behind the baffle and highest opposite the baffle with the centerline sample falling in the middle. The ranges for the average and maximum uranium isotopic activities are as follows:

1. Average U-234 from 85.7 (Zone 3) to 465 pCi/g (Zone 6); maximum U-234 from 89.6 (Zone 3) to 508 pCi/g (Zone 6)
2. Average U-235 from 4.88 (Zone 3) to 25.1 pCi/g (Zone 6); maximum U-235 from 5.66 (Zone 3) to 28.3 pCi/g (Zone 6)
3. Average U-238 from 0.72 (Zone 3) to 8.90 pCi/g (Zone 6); maximum U-238 from 0.883 (Zone 3) to 10.7 pCi/g (Zone 6).

For the remaining six samples from Zones 2, 4, 5, 7, and 8, the average and maximum uranium isotopic activities are as follows:

1. Average U-234 from 3,430 (Zone 8) to 19,400 pCi/g (Zone 5); maximum U-234 from 3,980 (Zone 8) to 27,500 pCi/g (Zone 5)
2. Average U-235 from 121 (Zone 8) to 1,010 pCi/g (Zone 5); maximum U-235 from 139 (Zone 8) to 1,250 pCi/g (Zone 5)
3. Average U-238 from 12.7 (Zone 8) to 930 pCi/g (Zone 5); maximum U-238 from 14.8 (Zone 8) to 1,330 pCi/g (Zone 4).

These results mirror the trend found in the upper zones with respect to U-234 activity being the highest and U-238 activity the lowest. However, now the lowest uranium content is found in the zone opposite the baffle and the highest uranium content in the zones behind the baffle.

Validation of the isotopic data focused on detector system calibrations and operational performance checks, laboratory method blank results, laboratory generated duplicate results, laboratory analytical yields, and laboratory control sample (LCS) results. The only parameter that resulted in application of a data qualifier is the LCS result. In the case of U-235, one of the three LCS results exceeded the prescribed recovery limits of 70 to 130% at 137.7%. As a result, all U-235 results associated with the high LCS recovery batch (i.e., Samples 1RD001, 1RD002, and 1RD003) are qualified as “J” indicating that these results may be biased high. Additionally, seven of the U-238 results are also qualified as “J” because the measured activities are only two to three times greater than their associated uncertainties. All other results are unqualified since they are more than three times the associated uncertainties.

### **G-4.3 TCLP Metal Results**

The laboratory-prepared composite samples (1RD01001TI and 1RD01002TI) were analyzed for arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver. Both composites were subjected to sample extraction as specified by SW-846 Method 1311, sample preparation and analysis of mercury as specified by SW-846 Method 7470A, sample preparation for the remaining analytes as specified by SW-846 Method 3015A, and sample analysis for these analytes as specified by SW-846 6010A.

The results are presented in Table G-3. Arsenic, lead, and selenium from Sample 1RD01001TI and silver in both samples were not detected (“U” code in Table G-3), and the results shown are the method detection limits (MDLs). Arsenic, lead, and selenium from Sample 1RD01002TI were detected but at levels less than 10 times the MDL (“B” code in Table G-3). The remaining metal analytes (barium, cadmium, chromium, and mercury) were detected in both samples at levels greater than 10 times the MDL. Sample 1RD01002TI exceeds the regulatory limits for “characteristic” hazardous waste for both cadmium and mercury. All other results are below the hazardous waste thresholds for these metals. However, cadmium and mercury results from both composite samples exceed the universal treatment standards (110 and 25 µg/L, respectively) that may be applicable to the treatment and/or disposal of the tank contents.

Data validation qualifiers are attached to eight of the metal analytical results. The barium concentrations in both samples are qualified with a “J” flag in Table G-3 to denote that the analyte is detectable at the reported values but that the values are only estimates due to low recoveries for the continuous calibration verification, matrix spike, and matrix spike duplicate. Cadmium concentrations in both samples are qualified with a “J” flag to denote that the analyte is detectable at the reported values but that the values are only estimates due to low recoveries for the matrix spike, matrix spike duplicate, and analytical spike. The silver concentrations in both samples are qualified with a “UJ” flag to denote that the analyte is non-detectable at the reported values but that the reported values are only estimates due to low recoveries for the matrix spike, matrix spike duplicate, and analytical spike. Finally, the arsenic and selenium concentrations for Sample 1RD01002TI are qualified with a “U” flag to denote that the sample results are greater than the MDLs, but less than five times the amount of analyte found in the preparation blanks.

### **G-4.4 Additional Isotopic Uranium Analyses**

In August 2001, BWXT Services, Inc., was requested to perform additional isotopic uranium analyses on the original Tank V-9 samples. For each of the original nine samples taken from Tank V-9, one of the five subsamples retained after the initial analysis was performed was randomly selected and fractions were removed for preparation by strong mineral acid dissolution and molten salt fusion. The resulting isotopic uranium analyses for both preparation methods are presented in Table G-4, along with

the associated uncertainty and relative percent difference. A duplicate fusion analysis was also performed on the Zone 2 sample (0107074-04D).

The same general trends are observed in the additional isotopic analyses that were present in the original analyses. The uppermost sampling zones (Zones 1, 3, and 6) containing primarily liquid display lower uranium activities than the bottom zones containing primarily sludge, with the lowest activity in the zone behind the baffle (Zone 3) and the highest activity in the zone opposite the baffle (Zone 6). Maximum U-234 activities in the upper zones range from 74.8 (Zone 3) to 567 pCi/g (Zone 6); maximum U-235 activities from 4.95 (Zone 3) to 30.9 pCi/g (Zone 6); and maximum U-238 activities from 0.95 (Zone 3) to 13.3 pCi/g (Zone 6).

In the bottom sampling zones, the trend is reversed with the lowest uranium activity found in the zone opposite the baffle (Zone 8) and the highest activity in the zone behind the baffle (Zone 5). Maximum U-234 activities range from 3,490 (Zone 8) to 56,500 pCi/g (Zone 5); maximum U-235 activities from 119 (Zone 8) to 2,520 pCi/g (Zone 5); and maximum U-238 activities from 10.5 (Zone 8) to 1,060 pCi/g (Zone 5).

Generally for any given sample, the dissolution and fusion isotopic uranium activities are in fairly close agreement. The relative percent differences in uranium activity between the two preparation methods vary from 1.1 to 24.8% for U-234, from 1.5 to 25.0% for U-235, and from 1.5 to 57.2% for U-238. Finally, the relative percent difference between the duplicate Zone 2 fusion analyses (1RD05102R9 and 0107074-04D) varied from 7.2% for U-234 to 79.7% for U-238.

## **G-5. SAMPLING LESSONS LEARNED**

The primary lesson learned during the sampling activity was that verbatim compliance with a written procedure might seem cumbersome when a difficulty is encountered, but timely execution of a change order to the procedure did not severely hamper the overall completion of the project. The approval process for changes to the procedures ensured that decisions were not made casually. ALARA and criticality concerns were given highest priority even when addressing a simple mechanical issue.

The video camera in the tank was most helpful. At the time the procedures were written, the reliability of the camera was underestimated. The only part of the procedure that required the camera was the initial reconnaissance and line-up of the glove bag with respect to the baffle. Beyond that, if the camera failed, the procedure could still be followed exactly; the equipment was designed to be operated without visual aid. Because the camera did survive in the radiation field, the images it provided gave an extra level of confidence to the sampling team. At each step, the visual confirmation reduced the need to proceed on faith.

The sludge encountered behind the baffle (Sampling Zones 3, 4 and 5) was substantially different from the training sludges used at the STAR Center. For future sampling activities, a wider range of practice material may better prepare the operators for unexpected conditions.

All of the sampling equipment was designed to fit into 55-gal drums for disposal. Some of the pieces, however, were packaged together in bundles that were too large to fit. Future equipment should use slightly smaller pieces to allow for packaging without exceeding the drum dimensions.

## G-6. REFERENCES

- Blackmore, 1998, Memo from C. S. Blackmore, INEEL, Power Burst Facility to J. T. Taylor, INEEL, Idaho Falls, "Criticality Safety Issues Associated with the TAN V-Tanks," CSB-004-98, March 10, 1998.
- BWXT Services, Inc. (Nuclear Environmental Laboratory Services), 2001a, "Data Reporting Package [Classical Analysis] for Bechtel BWXT Idaho, LLC, Sample Management Office," Report Number 0105041 I&MCA, May 29.
- BWXT Services, Inc. (Nuclear Environmental Laboratory Services), 2001b, "Data Reporting Package for Bechtel BWXT Idaho, LLC (BBWI), Sample Management Office," Report Number 0107074 RAD, August 8.
- DOE-ID, 1997, *Comprehensive Remedial Investigation/Feasibility Study for the Test Area North, Operable Unit 1-10 at the Idaho National Engineering and Environmental Laboratory*, Department of Energy Idaho Operations Office, DOE/ID-10557, Revision 0, November.
- DOE-ID, 2001, *Comprehensive Remedial Design/Remedial Action Work Plan for the Test Area North, Waste Area Group 1, Operable Unit 1-10, Group 2 Sites*, Department of Energy Idaho Operations Office, DOE/ID-10875, Rev. D, July.
- INEEL, 2001, *Voluntary Consent Order NEW-TAN-08 System Identification, TAN 616 Liquid Waste Treatment System*, Idaho National Engineering and Environmental Laboratory, INEEL/EXT-2000-01263, Revision 1, June.
- U.S. Environmental Protection Agency, *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846*, Third Edition, Final Update III, dated December 1996 (or most recent). (Cited as SW-846.)
- WESTON, 2001, V-Tank Remedial Action Technical Procedure, "Tank V-9 Sampling Procedure," Document No. A4J780.01, Revision 0, March 20.



## **Attachment G-1**

### **Raw Data and Data Validation Summaries**

Classical Analysis (Bulk Density and Percent Moisture) Data and Data Validation

Radionuclide Analysis Data and Data Validation

TCLP Metal Analysis Data and Data Validation

Additional Radionuclide Analysis Data and Data Validation

**Classical Analysis (Bulk Density and Percent Moisture)  
Data and Data Validation**

# MISCELLANEOUS CLASSICAL ANALYSIS DATA SHEET

**Lab Name:** BWXS-NEL Services      **Contract:** K97-180459  
**Lab Code:** BWLVA      **LTI#:** 0105041  
**TOS#:** ER-SOW-380 Rev 0      **SDG#:** 1RD001013A  
**Matrix:** Sludge      **Date Received:** 05/02/2001-05/09/2001  
**Analyte:** Bulk Density

CUSTOMER ID	RESULT	UNITS	C	Q	DETECTION LIMIT	LAB ID	V
1RD001013A	1.0	g/mL			NA	0105041-01	
1RD001023A	1.0	g/mL			NA	0105041-02	
1RD001033A	1.0	g/mL			NA	0105041-03	
1RD001043A	1.0	g/mL			NA	0105041-04	
1RD001053A	1.0	g/mL			NA	0105041-05	
1RD002013A	0.8	g/mL			NA	0105041-06	
1RD002023A	2.7	g/mL			NA	0105041-07	
1RD002033A	1.5	g/mL			NA	0105041-08	
1RD002043A	1.4	g/mL			NA	0105041-09	
1RD002053A	3.4	g/mL			NA	0105041-10	
1RD003013A	1.0	g/mL			NA	0105041-11	
1RD003023A	1.0	g/mL			NA	0105041-12	
1RD003033A	1.0	g/mL			NA	0105041-13	
1RD003043A	1.0	g/mL			NA	0105041-14	
1RD003053A	1.0	g/mL			NA	0105041-15	
1RD004013A	1.7	g/mL			NA	0105041-16	
1RD004023A	1.4	g/mL			NA	0105041-17	
1RD004033A	1.6	g/mL			NA	0105041-18	
1RD004043A	1.8	g/mL			NA	0105041-19	
1RD004053A	2.2	g/mL			NA	0105041-20	
1RD005013A	2.4	g/mL			NA	0105041-21	
1RD005023A	1.3	g/mL			NA	0105041-22	
1RD005033A	1.5	g/mL			NA	0105041-23	

Qualifiers: U = Not Detected. Result less than detection limit.

**COMMENTS:** All samples analyzed "as received".

# MISCELLANEOUS CLASSICAL ANALYSIS DATA SHEET

**Lab Name:** BWXS-NEL Services      **Contract:** K97-180459  
**Lab Code:** BWLVA      **LTi#:** 0105041  
**TOS#:** ER-SOW-380 Rev 0      **SDG#:** 1RD001013A  
**Matrix:** Sludge      **Date Received:** 05/02/2001-05/09/2001  
**Analyte:** Bulk Density

CUSTOMER ID	RESULT	UNITS	C	Q	DETECTION LIMIT	LAB ID	V
1RD005043A	1.5	g/mL			NA	0105041-24	
1RD005053A	1.9	g/mL			NA	0105041-25	
1RD006013A	1.5	g/mL			NA	0105041-26	
1RD006023A	1.6	g/mL			NA	0105041-27	
1RD006033A	1.8	g/mL			NA	0105041-28	
1RD006043A	1.6	g/mL			NA	0105041-29	
1RD006053A	1.9	g/mL			NA	0105041-30	
1RD007013A	1.0	g/mL			NA	0105041-31	
1RD007023A	1.1	g/mL			NA	0105041-32	
1RD007033A	1.0	g/mL			NA	0105041-33	
1RD007043A	1.1	g/mL			NA	0105041-34	
1RD007053A	1.0	g/mL			NA	0105041-35	
1RD008013A	1.4	g/mL			NA	0105041-36	
1RD008023A	1.3	g/mL			NA	0105041-37	
1RD008033A	1.3	g/mL			NA	0105041-38	
1RD008043A	1.2	g/mL			NA	0105041-39	
1RD008053A	1.3	g/mL			NA	0105041-40	
1RD009013A	1.6	g/mL			NA	0105041-41	
1RD009023A	2.3	g/mL			NA	0105041-42	
1RD009033A	1.1	g/mL			NA	0105041-43	
1RD009043A	1.7	g/mL			NA	0105041-44	
1RD009053A	1.2	g/mL			NA	0105041-45	

Qualifiers: U = Not Detected. Result less than detection limit.

**COMMENTS:** All samples analyzed "as received".  
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# MISCELLANEOUS CLASSICAL ANALYSIS DATA SHEET

**Lab Name:** BWXS-NEL Services      **Contract:** K97-180459  
**Lab Code:** BWLVA      **LTI#:** 0105041  
**TOS#:** ER-SOW-380 Rev 0      **SDG#:** 1RD001013A  
**Matrix:** Sludge      **Date Received:** 05/02/2001-05/09/2001  
**Analyte:** Percent Moisture

CUSTOMER ID	RESULT	UNITS	C	Q	DETECTION LIMIT	LAB ID	V
1RD001013A	98.0	%			NA	0105041-01	
1RD001023A	98.3	%			NA	0105041-02	
1RD001033A	97.9	%			NA	0105041-03	
1RD001043A	98.0	%			NA	0105041-04	
1RD001053A	98.1	%			NA	0105041-05	
1RD002013A	46.9	%			NA	0105041-06	
1RD002023A	46.7	%			NA	0105041-07	
1RD002033A	46.1	%			NA	0105041-08	
1RD002043A	46.3	%			NA	0105041-09	
1RD002053A	47.6	%			NA	0105041-10	
1RD003013A	97.4	%			NA	0105041-11	
1RD003023A	97.1	%			NA	0105041-12	
1RD003033A	93.2	%			NA	0105041-13	
1RD003043A	100.4	%			NA	0105041-14	
1RD003053A	97.0	%			NA	0105041-15	
1RD004013A	52.5	%			NA	0105041-16	
1RD004023A	53.1	%			NA	0105041-17	
1RD004033A	43.7	%			NA	0105041-18	
1RD004043A	54.8	%			NA	0105041-19	
1RD004053A	39.5	%			NA	0105041-20	
1RD005013A	55.7	%			NA	0105041-21	
1RD005023A	58.5	%			NA	0105041-22	
1RD005033A	64.7	%			NA	0105041-23	

Qualifiers: U = Not Detected. Result less than detection limit.

**COMMENTS:** All samples analyzed "as received".

# MISCELLANEOUS CLASSICAL ANALYSIS DATA SHEET

**Lab Name:** BWXS-NEL Services      **Contract:** K97-180459  
**Lab Code:** BWLVA      **LTI#:** 0105041  
**TOS#:** ER-SOW-380 Rev 0      **SDG#:** 1RD001013A  
**Matrix:** Sludge      **Date Received:** 05/02/2001-05/09/2001  
**Analyte:** Percent Moisture

CUSTOMER ID	RESULT	UNITS	C	Q	DETECTION LIMIT	LAB ID	V
1RD005043A	55.9	%			NA	0105041-24	
1RD005053A	55.2	%			NA	0105041-25	
1RD006013A	43.6	%			NA	0105041-26	
1RD006023A	39.6	%			NA	0105041-27	
1RD006033A	37.0	%			NA	0105041-28	
1RD006043A	40.8	%			NA	0105041-29	
1RD006053A	41.0	%			NA	0105041-30	
1RD007013A	94.0	%			NA	0105041-31	
1RD007023A	94.3	%			NA	0105041-32	
1RD007033A	93.9	%			NA	0105041-33	
1RD007043A	94.4	%			NA	0105041-34	
1RD007053A	94.3	%			NA	0105041-35	
1RD008013A	66.9	%			NA	0105041-36	
1RD008023A	67.5	%			NA	0105041-37	
1RD008033A	66.4	%			NA	0105041-38	
1RD008043A	66.5	%			NA	0105041-39	
1RD008053A	67.1	%			NA	0105041-40	
1RD009013A	62.0	%			NA	0105041-41	
1RD009023A	62.4	%			NA	0105041-42	
1RD009033A	61.6	%			NA	0105041-43	
1RD009043A	59.9	%			NA	0105041-44	
1RD009053A	63.4	%			NA	0105041-45	

Qualifiers: U = Not Detected. Result less than detection limit.

**COMMENTS:** All samples analyzed "as received".

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# ORIGINAL

## INORGANIC AND MISCELLANEOUS CLASSICAL ANALYSES LIMITATIONS AND VALIDATION REPORT

### 1. TASK SPECIFIC VALIDATION IDENTIFICATION INFORMATION

<u>L&amp;V Report Number:</u>	DNT-104-01	<b>RECEIVED</b> JUN 12 2001 ENVIRONMENTAL RESTORATION DEPT.
<u>SDG Number:</u>	1RD001013A	
<u>SDG Type:</u>	3	
<u>Number of Samples:</u>	9	
<u>Sample Matrix:</u>	Sludge	
<u>Applicable Analytes:</u>	Bulk Density and Total Moisture	
<u>Reporting Tier:</u>	1	
<u>TOS Number:</u>	ER-SOW-380R1	
<u>TOS Title:</u>	Analyses of Samples Collected for the Tank V-9 (TSF-18) Sampling for Operable Unit 1-10 Project , Document No. ER-SOW-380R1	
<u>Analytical Laboratory:</u>	BWX Technologies, Inc. (BWXT)	
<u>LTI Number:</u>	0105041	
<u>Data Validator:</u>	David N. Thompson	
<u>Validator Affiliation:</u>	INEEL SMO	
<u>Validation Level:</u>	A	
<u>Completion Date:</u>	06/12/01	

## 2. IDV PRECAUTIONS AND LIMITATIONS

General precautions and limitations associated with inorganic and miscellaneous classical analyses analytical method data validation (IDV) are delineated in Section 2 of Technical Procedure (TPR)-132 (Reference 1) and are included in this Limitations and Validation (L&V) report as Attachment 5.

## 3. INTRODUCTION

A Level-A IDV [see Guide Document (GDE)-7003 (Reference 2)], following the procedures outlined in TPR-132, was performed on the inorganic and miscellaneous classical analyses data package (IDP), identified as sample delivery group (SDG) number 1RD001013A that was compiled by BWXT. TPR-132 is an Idaho National Engineering and Environmental Laboratory (INEEL) Sample Management Office (SMO) document that has revised the validation procedures outlined in the United States Environmental Protection Agency (USEPA) Functional Guidelines (Reference 3) to more aptly apply to IDPs prepared in accordance with the generic inorganic and miscellaneous classical analyses (I&MCA) statement of work [see ER-SOW-156 (Reference 4)] routinely requested by the INEEL SMO. Nine (9) sludge samples were collected for the Tank V-9 [Technical Support Facility (TSF)-18] Sampling for the Operable Unit (OU) 1-10 Project. BWXT was contracted to analyze the 9 sludge samples for Bulk Density and Total Moisture using the applicable ASTM Standard D5057-90 (Reference 5) and ASTM Standard E949-88 (Reference 6), respectively, in conjunction with both the task order statement of work [ER-SOW-380 (Reference 7)] and ER-SOW-156. Level-A IDV includes a data confirmation (see definition in Section 8) step.

## 4. SAMPLE IDENTIFICATION

According to ER-SOW-380 and each associated chain-of-custody (COC) form, BWXT was required to subdivide, or aliquot, each of the 9 sludge samples into five subsamples prior to analysis. The following table identifies and describes that relationship:

FIELD SAMPLE IDENTIFICATION NUMBER	ALIQOT OR SUBSAMPLE IDENTIFICATION NUMBER	LABORATORY IDENTIFICATION NUMBER	ORIGINAL SAMPLE MATRIX
1RD0010	1RD001013A	0105041-01	Sludge
	1RD001023A	0105041-02	Sludge
	1RD001033A	0105041-03	Sludge
	1RD001043A	0105041-04	Sludge
	1RD001053A	0105041-05	Sludge



FIELD SAMPLE IDENTIFICATION NUMBER	ALIQOT OR SUBSAMPLE IDENTIFICATION NUMBER	LABORATORY IDENTIFICATION NUMBER	ORIGINAL SAMPLE MATRIX
1RD0020	1RD002013A	0105041-06	Sludge
	1RD002023A	0105041-07	Sludge
	1RD002033A	0105041-08	Sludge
	1RD002043A	0105041-09	Sludge
	1RD002053A	0105041-10	Sludge
1RD0030	1RD003013A	0105041-11	Sludge
	1RD003023A	0105041-12	Sludge
	1RD003033A	0105041-13	Sludge
	1RD003043A	0105041-14	Sludge
	1RD003053A	0105041-15	Sludge
1RD0040	1RD004013A	0105041-16	Sludge
	1RD004023A	0105041-17	Sludge
	1RD004033A	0105041-18	Sludge
	1RD004043A	0105041-19	Sludge
	1RD004053A	0105041-20	Sludge
1RD0050	1RD005013A	0105041-21	Sludge
	1RD005023A	0105041-22	Sludge
	1RD005033A	0105041-23	Sludge
	1RD005043A	0105041-24	Sludge
	1RD005053A	0105041-25	Sludge
1RD0060	1RD006013A	0105041-26	Sludge
	1RD006023A	0105041-27	Sludge
	1RD006033A	0105041-28	Sludge
	1RD006043A	0105041-29	Sludge
	1RD006053A	0105041-30	Sludge
1RD0070	1RD007013A	0105041-31	Sludge
	1RD007023A	0105041-32	Sludge
	1RD007033A	0105041-33	Sludge
	1RD007043A	0105041-34	Sludge
	1RD007053A	0105041-35	Sludge
1RD0080	1RD008013A	0105041-36	Sludge
	1RD008023A	0105041-37	Sludge
	1RD008033A	0105041-38	Sludge
	1RD008043A	0105041-39	Sludge
	1RD008053A	0105041-40	Sludge
1RD0090	1RD009013A	0105041-41	Sludge
	1RD009023A	0105041-42	Sludge
	1RD009033A	0105041-43	Sludge
	1RD009043A	0105041-44	Sludge
	1RD009053A	0105041-45	Sludge

## 5. CONTRACT AND TECHNICAL REVIEW (CTR) COMMENTS

1. According to ER-SOW-380R1 and the associated COCs, the laboratory was required to subdivide, or aliquot, each of the 9 sludge samples into 5 subsamples. Due to the geophysical nature of the two analytical methods (bulk density and total moisture), the only applicable quality control parameter is the sample

duplicate. A SDG Type-3 Form #7 (Duplicate form) was not provided for this data. Although sample duplicate data were not typically reported, the laboratory essentially performed a duplicate for every sample, 5 fold. As mentioned in the Introduction in Section 3.0, this validation was performed by following the procedures outlined in TPR-132 and therefore, according to Section 4.3.11.5.3 for nonaqueous samples, the sample results were assessed based on their subsample's relative percent differences (RPDs) to each other. For each sample, the highest and lowest value reported from the five aliquots were used for this calculation. If the RPD was greater than 35% but less than or equal to 50%, the associated sample results were qualified with a "J" validation flag. If the RPD was greater than 50%, the associated sample results were qualified with a "R" validation flag.

Bulk Density results for sample 1RD0040 are qualified with a "J" validation flag due to a RPD of 44.4%. Bulk Density results for samples 1RD0020, 1RD0050, and 1RD0090 are qualified with a "R" validation flag due to RPDs of 123.8%, 59.4%, and 70.6%, respectively.

Total Moisture results were within the duplicate sample control limits and do not require qualification.

## 6.0 DATA LIMITATION OVERVIEW

### 6.1 Summary of Qualified Data

Sample 1RD0040 received a "J" validation qualifying flag for Bulk Density to denote that the data is detectable at the reported value but the reported value is only an estimate due to anomalies in the quality control data. Samples 1RD0020, 1RD0050, and 1RD0090 received "R" validation qualifying flags for Bulk Density to denote that the accuracy of the data is so questionable that it is recommended the data not be used. (see CTR comment #1)

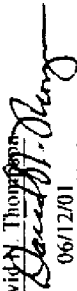
### 6.2 Data Confirmation Summary

FIELD SAMPLE DATA POINTS ASSOCIATED WITH SDG# 1RD001013A				
Total Number	Number Confirmed	Number Confirmed to be Correctly Reported	Number Confirmed to be Falsely Reported	Actual Proportion Falsely Reported (%)
9	9	9	0	0

### 6.3 Data Assessment Sheet (page 1 of 2)

L&V Report#: DNT-104-01  
 SDG #: IRD001013A  
 TOS #: ER-SOW-380R1  
 SOW #: ER-SOW-156  
 LTI #: 0105041

Validation Level: A  
 SDG Type: 3  
 Reporting Tier: 1  
 Samples by Matrix: Sludge (9 samples)  
 Laboratory: BWX Technologies, Inc.

Assessor's Affiliation: INEEL SMO  
 Assessor's Name: David L. Thompson  
 Assessor's Signature:   
 Assessment Date: 06/12/01

#### DATA ASSESSMENT SUMMARY TABLE

ITEM #	ASSESSMENT ITEM	ANALYTES	
		Bulk Density	Total Density
1.	Holding Time	0	0
2.	AQS Calibration	N/A	N/A
3.	ICV	N/A	N/A
4.	CCVs	N/A	N/A
5.	LLC Standards	N/A	N/A
6.	ICBs/CCIBs	N/A	N/A
7.	Preparation Blank	N/A	N/A
8.	ICP ICSs	N/A	N/A
9.	Matrix Spike	N/A	N/A
10.	Analytical Spike	N/A	N/A
11.	Laboratory Duplicate	0	0
12.	MSD	N/A	N/A
13.	ICS	N/A	N/A
14.	MSA	N/A	N/A
15.	Serial Dilution	N/A	N/A
16.	LRA Standard	N/A	N/A
17.	CRC Standard	N/A	N/A
18.	Data Confirmation	0	0
19.	Miscellaneous Items	0	0
20.	Overall Assessment	0	0

**6.3 Data Assessment Sheet (page 2 of 2)**

**DATA ASSESSMENT CODES**

- O = Assessment item was within applicable control limits and, if considered alone, would not cause any data to be assigned a "UJ," "J," or "R" validation flag.
- M = Assessment item was outside applicable control limits and, if considered alone, would: (a) cause one or more field sample data points to be assigned either a "UJ" or "J" validation flag, but (b) not cause any data to be assigned a "R" validation flag.
- Z = Assessment item was outside applicable control limits and, if considered alone, would cause one or more field sample data points to be assigned a "R" validation flag.
- N/A = Assessment item is not applicable.
- NP = Assessment item was required but was not performed and/or documented by the laboratory.
- X = Contractual and/or technical anomalies were noted but, based on the professional judgement of the assessor, none of the associated data were adversely affected.
- I = Contractual and/or technical anomalies were noted and, based on the professional judgement of the assessor, at least a portion of the data were adversely affected and/or could not be properly assessed. As a result, at least one applicable field sample data point was qualified with either a "UJ," "J," or "R" validation flag.
- G = The units reported for at least one applicable field sample data point did not correlate with the test method employed.

COMMENTS: None.

#### 6.4 Data Validation Flag Table

SDG # 1RD001013A		
SAMPLE ID	ANALYTES	
	Bulk Density	Total Moisture
1RD0010		
1RD0020	R	
1RD0030		
1RD0040	J	
1RD0050	R	
1RD0060		
1RD0070		
1RD0080		
1RD0090	R	

#### DEFINITIONS OF DATA VALIDATION FLAGS

- U = The material was analyzed for and was detected at or above the applicable detection limit. However, the associated value was less than 5 times the highest positive amount in any laboratory blank. In most instances a "U" validation flag will be accompanied by a "B" laboratory flag.
- UJ = The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise. A "UJ" validation flag is not differentiated from the combined action of both a "U" and "J" validation flag.
- J = The material was analyzed for and was detected at or above the applicable detection limit. The associated value is an estimate and may be inaccurate or imprecise.
- R = The accuracy of the data is so questionable that it is recommended the data not be used. For any given data point, a "R" validation flag overrides all other applicable validation flags.

#### 6.5 Summary of Data Usability

There were 9 field sample data points associated with the SDG #1RD001013A IDP. Of these field sample data points: (a) 5 were assessed and left unqualified, (b) 1 was assessed and qualified with a "J" validation flag, and (c) 3 were assessed and qualified with a "R" validation flag. Using the criteria outlined in EPA540-R-93-071 (Reference 8): (1) the 5 field sample data points (55.6% of the total) that were assessed and left unqualified can be categorized as definitive data with no associated quality control deficiencies, (2) the 1 data

point (11.1% of the total) that was assessed and qualified with a "J" validation flag can be categorized as definitive data with a positively identified analyte concentration that is only an estimate due to quality control deficiencies, and (3) the 3 field data points (33.3% of the total) that were assessed and qualified with a "R" validation flag can be categorized as definitive data whose accuracy is so questionable that it is recommended the data not be used.

## **7. FLAGRANT CONTRACTUAL DEFICIENCIES**

### **7.1 Missed Holding Times**

None.

### **7.2 Use of Unauthorized Methods**

None.

### **7.3 Other**

None.

## **Radionuclide Analysis Data and Data Validation**





## RADIONUCLIDE ANALYSIS RESULTS

Date: May 29, 2001

Lab Name: BWLVA

Case No.: ER-SOW-380

Report No.: 0105041

SDG No.: 1RD001013A

BBWI ID	Lab ID	Sample Matrix	Anal Type	Sample Value	Sample Uncer	Units	Anal Date	Sample Date	Sample Size	Yield	Det ID	MDA
1RD001013A	0105041-01	Sludge	U-234	1.90E+02	1.58E+01	pCi/g	05/25/01	04/30/01	0.8088	57.2392	A06	5.09E-01
1RD001013A	0105041-01	Sludge	U-235	9.94E+00	1.36E+00	pCi/g	05/25/01	04/30/01	0.8088	57.2392	A06	3.29E-01
1RD001013A	0105041-01	Sludge	U-238	1.53E+00	4.62E-01	pCi/g	05/25/01	04/30/01	0.8088	57.2	A06	3.29E-01
1RD001023A	0105041-02	Sludge	U-234	1.73E+02	1.44E+01	pCi/g	05/25/01	04/30/01	0.8050	54.8	A07	6.51E-01
1RD001023A	0105041-02	Sludge	U-235	8.98E+00	1.25E+00	pCi/g	05/25/01	04/30/01	0.8050	54.8	A07	4.36E-01
1RD001023A	0105041-02	Sludge	U-238	1.39E+00	4.29E-01	pCi/g	05/25/01	04/30/01	0.8050	54.8	A07	3.12E-01
1RD001033A	0105041-03	Sludge	U-234	1.91E+02	1.60E+01	pCi/g	05/25/01	04/30/01	0.8040	57.9	A08	5.08E-01
1RD001033A	0105041-03	Sludge	U-235	1.01E+01	1.42E+00	pCi/g	05/25/01	04/30/01	0.8040	57.9	A08	3.64E-01
1RD001033A	0105041-03	Sludge	U-238	1.69E+00	5.11E-01	pCi/g	05/25/01	04/30/01	0.8040	57.9	A08	3.64E-01
1RD001043A	0105041-04	Sludge	U-234	1.69E+02	1.41E+01	pCi/g	05/25/01	04/30/01	0.8080	52.8	A18	5.19E-01
1RD001043A	0105041-04	Sludge	U-235	9.49E+00	1.31E+00	pCi/g	05/25/01	04/30/01	0.8080	52.8	A18	3.90E-01
1RD001043A	0105041-04	Sludge	U-238	7.14E-01	3.15E-01	pCi/g	05/25/01	04/30/01	0.8080	52.8	A18	3.21E-01
1RD001053A	0105041-05	Sludge	U-234	1.65E+02	1.36E+01	pCi/g	05/25/01	04/30/01	0.8251	63.8	A19	2.56E-01
1RD001053A	0105041-05	Sludge	U-235	9.94E+00	1.25E+00	pCi/g	05/25/01	04/30/01	0.8251	63.8	A19	2.56E-01
1RD001053A	0105041-05	Sludge	U-238	1.28E+00	3.73E-01	pCi/g	05/25/01	04/30/01	0.8251	63.8	A19	2.56E-01
1RD002013A	0105041-06	Sludge	U-234	1.55E+04	1.25E+03	pCi/g	05/25/01	04/30/01	0.0208	78.7	A20	1.19E+01
1RD002013A	0105041-06	Sludge	U-235	6.05E+02	6.60E+01	pCi/g	05/25/01	04/30/01	0.0208	78.7	A20	1.10E+01
1RD002013A	0105041-06	Sludge	U-238	1.33E+02	2.39E+01	pCi/g	05/25/01	04/30/01	0.0208	78.7	A20	1.10E+01
1RD002023A	0105041-07	Sludge	U-234	5.33E+03	4.46E+02	pCi/g	05/25/01	04/30/01	0.0208	74.7	A22	1.10E+01
1RD002023A	0105041-07	Sludge	U-235	2.31E+02	3.45E+01	pCi/g	05/25/01	04/30/01	0.0208	74.7	A22	9.85E+00
1RD002023A	0105041-07	Sludge	U-238	1.14E+02	2.26E+01	pCi/g	05/25/01	04/30/01	0.0208	74.7	A22	9.85E+00
1RD002033A	0105041-08	Sludge	U-234	6.93E+03	5.70E+02	pCi/g	05/25/01	04/30/01	0.0213	78.6	A23	8.48E+00
1RD002033A	0105041-08	Sludge	U-235	2.55E+02	3.49E+01	pCi/g	05/25/01	04/30/01	0.0213	78.6	A23	8.48E+00
1RD002033A	0105041-08	Sludge	U-238	1.11E+02	2.08E+01	pCi/g	05/25/01	04/30/01	0.0213	78.6	A23	8.48E+00
1RD002043A	0105041-09	Sludge	U-234	1.12E+04	9.08E+02	pCi/g	05/25/01	04/30/01	0.0221	72.1	A24	8.85E+00
1RD002043A	0105041-09	Sludge	U-235	3.34E+02	4.26E+01	pCi/g	05/25/01	04/30/01	0.0221	72.1	A24	8.85E+00
1RD002043A	0105041-09	Sludge	U-238	1.36E+02	2.40E+01	pCi/g	05/25/01	04/30/01	0.0221	72.1	A24	8.85E+00
1RD002053A	0105041-10	Sludge	U-234	8.65E+03	7.30E+02	pCi/g	05/28/01	04/30/01	0.0239	80.7	A03	1.13E+01
1RD002053A	0105041-10	Sludge	U-235	3.85E+02	4.78E+01	pCi/g	05/28/01	04/30/01	0.0239	80.7	A03	9.25E+00
1RD002053A	0105041-10	Sludge	U-238	1.00E+02	2.05E+01	pCi/g	05/28/01	04/30/01	0.0239	80.7	A03	9.25E+00
1RD003013A	0105041-11	Sludge	U-234	8.42E+01	6.94E+00	pCi/g	05/28/01	05/01/01	2.0319	76.7	A04	1.46E-01
1RD003013A	0105041-11	Sludge	U-235	4.49E+00	5.52E-01	pCi/g	05/28/01	05/01/01	2.0319	76.7	A04	1.62E-01
1RD003013A	0105041-11	Sludge	U-238	6.69E-01	1.81E-01	pCi/g	05/28/01	05/01/01	2.0319	76.7	A04	1.69E-01
1RD003023A	0105041-12	Sludge	U-234	8.96E+01	7.34E+00	pCi/g	05/28/01	05/01/01	2.0328	72.6	A05	1.35E-01
1RD003023A	0105041-12	Sludge	U-235	5.22E+00	6.09E-01	pCi/g	05/28/01	05/01/01	2.0328	72.6	A05	1.03E-01
1RD003023A	0105041-12	Sludge	U-238	8.31E-01	1.93E-01	pCi/g	05/28/01	05/01/01	2.0328	72.6	A05	1.03E-01
1RD003033A	0105041-13	Sludge	U-234	8.57E+01	7.02E+00	pCi/g	05/28/01	05/01/01	2.0310	79.7	A06	1.46E-01
1RD003033A	0105041-13	Sludge	U-235	5.66E+00	6.33E-01	pCi/g	05/28/01	05/01/01	2.0310	79.7	A06	9.43E-02

000019

FORM I

## BBWL SAMPLE MANAGEMENT OFFICE

## RADIONUCLIDE ANALYSIS RESULTS

Date: May 29, 2001

Lab Name:

BWLVA

Case No.:

ER-SOW-380

Report No.:

0105041

SDG No.:

1RD001013A

BBWL ID	Lab ID	Sample Matrix	Anal Type	Sample Value	Sample Uncer	Units	Anal Date	Sample Date	Sample Size	Yield	Det ID	MDA
1RD003033A	0105041-13	Sludge	U-238	8.83E-01	1.92E-01	pCi/g	05/28/01	05/01/01	2.0310	79.7	A06	9.43E-02
1RD003043A	0105041-14	Sludge	U-234	8.82E+01	7.04E+00	pCi/g	05/28/01	05/01/01	2.0390	67.8	A07	2.08E-01
1RD003043A	0105041-14	Sludge	U-235	4.83E+00	5.71E-01	pCi/g	05/28/01	05/01/01	2.0390	67.8	A07	1.39E-01
1RD003043A	0105041-14	Sludge	U-238	5.91E-01	1.58E-01	pCi/g	05/28/01	05/01/01	2.0390	67.8	A07	9.96E-02
1RD003053A	0105041-15	Sludge	U-234	8.26E+01	6.80E+00	pCi/g	05/28/01	05/01/01	2.0225	87.3	A08	1.34E-01
1RD003053A	0105041-15	Sludge	U-235	4.21E+00	5.14E-01	pCi/g	05/28/01	05/01/01	2.0225	87.3	A08	9.60E-02
1RD003053A	0105041-15	Sludge	U-238	6.24E-01	1.60E-01	pCi/g	05/28/01	05/01/01	2.0225	87.3	A08	9.60E-02
1RD004013A	0105041-16	Sludge	U-234	1.38E+04	1.12E+03	pCi/g	05/28/01	05/02/01	0.0235	77.8	A22	9.32E+00
1RD004013A	0105041-16	Sludge	U-235	7.19E+02	7.42E+01	pCi/g	05/28/01	05/02/01	0.0235	77.8	A22	8.35E+00
1RD004013A	0105041-16	Sludge	U-238	3.66E+02	4.47E+01	pCi/g	05/28/01	05/02/01	0.0235	77.8	A22	8.35E+00
1RD004023A	0105041-17	Sludge	U-234	2.22E+04	1.78E+03	pCi/g	05/28/01	05/02/01	0.0202	82.3	A23	8.54E+00
1RD004023A	0105041-17	Sludge	U-235	8.98E+02	8.91E+01	pCi/g	05/28/01	05/02/01	0.0202	82.3	A23	8.54E+00
1RD004023A	0105041-17	Sludge	U-238	3.08E+02	3.98E+01	pCi/g	05/28/01	05/02/01	0.0202	82.3	A23	8.54E+00
1RD004033A	0105041-18	Sludge	U-234	7.44E+03	6.22E+02	pCi/g	05/29/01	05/02/01	0.0219	73.0	A03	1.36E+01
1RD004033A	0105041-18	Sludge	U-235	3.54E+02	4.78E+01	pCi/g	05/29/01	05/02/01	0.0219	73.0	A03	1.12E+01
1RD004033A	0105041-18	Sludge	U-238	3.03E+02	4.31E+01	pCi/g	05/29/01	05/02/01	0.0219	73.0	A03	1.12E+01
1RD004043A	0105041-19	Sludge	U-234	1.86E+04	1.51E+03	pCi/g	05/28/01	05/02/01	0.0200	72.0	A04	1.58E+01
1RD004043A	0105041-19	Sludge	U-235	7.57E+02	8.29E+01	pCi/g	05/28/01	05/02/01	0.0200	72.0	A04	1.75E+01
1RD004043A	0105041-19	Sludge	U-238	3.78E+02	5.04E+01	pCi/g	05/28/01	05/02/01	0.0200	72.0	A04	1.83E+01
1RD004053A	0105041-20	Sludge	U-234	8.41E+03	6.92E+02	pCi/g	05/28/01	05/02/01	0.0206	75.6	A05	1.28E+01
1RD004053A	0105041-20	Sludge	U-235	3.74E+02	4.75E+01	pCi/g	05/28/01	05/02/01	0.0206	75.6	A05	9.75E+00
1RD004053A	0105041-20	Sludge	U-238	2.38E+02	3.51E+01	pCi/g	05/28/01	05/02/01	0.0206	75.6	A05	9.75E+00
1RD005013A	0105041-21	Sludge	U-234	7.03E+03	5.82E+02	pCi/g	05/28/01	05/03/01	0.0210	75.4	A06	1.49E+01
1RD005013A	0105041-21	Sludge	U-235	4.00E+02	4.96E+01	pCi/g	05/28/01	05/03/01	0.0210	75.4	A06	9.64E+00
1RD005013A	0105041-21	Sludge	U-238	4.14E+02	5.08E+01	pCi/g	05/28/01	05/03/01	0.0210	75.4	A06	9.64E+00
1RD005023A	0105041-22	Sludge	U-234	2.16E+04	1.74E+03	pCi/g	05/28/01	05/03/01	0.0211	69.6	A07	1.96E+01
1RD005023A	0105041-22	Sludge	U-235	1.22E+03	1.16E+02	pCi/g	05/28/01	05/03/01	0.0211	69.6	A07	1.31E+01
1RD005023A	0105041-22	Sludge	U-238	1.05E+03	1.03E+02	pCi/g	05/28/01	05/03/01	0.0211	69.6	A07	9.30E+00
1RD005033A	0105041-23	Sludge	U-234	8.21E+03	6.74E+02	pCi/g	05/28/01	05/03/01	0.0259	86.6	A08	1.06E+01
1RD005033A	0105041-23	Sludge	U-235	4.78E+02	5.30E+01	pCi/g	05/28/01	05/03/01	0.0259	86.6	A08	7.56E+00
1RD005033A	0105041-23	Sludge	U-238	4.28E+02	4.88E+01	pCi/g	05/28/01	05/03/01	0.0259	86.6	A08	7.56E+00
1RD005043A	0105041-24	Sludge	U-234	1.32E+04	1.06E+03	pCi/g	05/28/01	05/03/01	0.0225	76.9	A18	1.28E+01
1RD005043A	0105041-24	Sludge	U-235	7.77E+02	7.77E+01	pCi/g	05/28/01	05/03/01	0.0225	76.9	A18	9.61E+00
1RD005043A	0105041-24	Sludge	U-238	5.61E+02	6.03E+01	pCi/g	05/28/01	05/03/01	0.0225	76.9	A18	7.89E+00
1RD005053A	0105041-25	Sludge	U-234	2.01E+04	1.61E+03	pCi/g	05/28/01	05/03/01	0.0234	70.4	A19	8.18E+00
1RD005053A	0105041-25	Sludge	U-235	1.14E+03	1.08E+02	pCi/g	05/28/01	05/03/01	0.0234	70.4	A19	8.18E+00
1RD005053A	0105041-25	Sludge	U-238	1.33E+03	1.23E+02	pCi/g	05/28/01	05/03/01	0.0234	70.4	A19	8.18E+00
1RD006013A	0105041-26	Sludge	U-234	2.38E+04	1.99E+03	pCi/g	05/28/01	05/08/01	0.0085	78.2	A20	2.92E+01

000020

## FORM 1

## BBWL SAMPLE MANAGEMENT OFFICE

## RADIONUCLIDE ANALYSIS RESULTS

Date: May 29, 2001

Lab Name:

BWLVA

Case No.:

ER-SOW-380

Report No.:

0105041

SDG No.:

1RD001013A

BBWL ID	Lab ID	Sample Matrix	Anal Type	Sample Value	Sample Uncer	Units	Anal Date	Sample Date	Sample Size	Yield	Det ID	MDA
1RD006013A	0105041-26	Sludge	U-235	1.13E+03	1.34E+02	pCi/g	05/28/01	05/08/01	0.0085	78.2	A20	2.72E+01
1RD006013A	0105041-26	Sludge	U-238	8.18E+02	1.06E+02	pCi/g	05/28/01	05/08/01	0.0085	78.2	A20	2.72E+01
1RD006023A	0105041-27	Sludge	U-234	2.75E+04	2.30E+03	pCi/g	05/28/01	05/08/01	0.0083	78.7	A22	2.61E+01
1RD006023A	0105041-27	Sludge	U-235	1.25E+03	1.46E+02	pCi/g	05/28/01	05/08/01	0.0083	78.7	A22	2.34E+01
1RD006023A	0105041-27	Sludge	U-238	9.72E+02	1.22E+02	pCi/g	05/28/01	05/08/01	0.0083	78.7	A22	2.34E+01
1RD006033A	0105041-28	Sludge	U-234	1.73E+04	1.46E+03	pCi/g	05/28/01	05/08/01	0.0084	79.8	A23	2.12E+01
1RD006033A	0105041-28	Sludge	U-235	1.11E+03	1.31E+02	pCi/g	05/28/01	05/08/01	0.0084	79.8	A23	2.12E+01
1RD006033A	0105041-28	Sludge	U-238	1.19E+03	1.37E+02	pCi/g	05/28/01	05/08/01	0.0084	79.8	A23	2.12E+01
1RD006043A	0105041-29	Sludge	U-234	1.46E+04	1.23E+03	pCi/g	05/28/01	05/08/01	0.0084	77.0	A24	1.95E+01
1RD006043A	0105041-29	Sludge	U-235	7.47E+02	9.57E+01	pCi/g	05/28/01	05/08/01	0.0084	77.0	A24	1.95E+01
1RD006043A	0105041-29	Sludge	U-238	9.30E+02	1.12E+02	pCi/g	05/28/01	05/08/01	0.0084	77.0	A24	1.95E+01
1RD006053A	0105041-30	Sludge	U-234	1.37E+04	1.17E+03	pCi/g	05/29/01	05/09/01	0.0088	71.1	A07	4.59E+01
1RD006053A	0105041-30	Sludge	U-235	8.21E+02	1.06E+02	pCi/g	05/29/01	05/09/01	0.0088	71.1	A07	3.07E+01
1RD006053A	0105041-30	Sludge	U-238	7.41E+02	9.87E+01	pCi/g	05/29/01	05/09/01	0.0088	71.1	A07	2.20E+01
1RD007013A	0105041-31	Sludge	U-234	5.04E+02	4.14E+01	pCi/g	05/29/01	05/03/01	0.4124	85.5	A08	6.70E-01
1RD007013A	0105041-31	Sludge	U-235	2.65E+01	3.04E+00	pCi/g	05/29/01	05/03/01	0.4124	85.5	A08	4.80E-01
1RD007013A	0105041-31	Sludge	U-238	1.07E+01	1.63E+00	pCi/g	05/29/01	05/03/01	0.4124	85.5	A08	4.80E-01
1RD007023A	0105041-32	Sludge	U-234	4.80E+02	3.91E+01	pCi/g	05/29/01	05/03/01	0.4046	82.0	A18	6.66E-01
1RD007023A	0105041-32	Sludge	U-235	2.83E+01	3.06E+00	pCi/g	05/29/01	05/03/01	0.4046	82.0	A18	5.01E-01
1RD007023A	0105041-32	Sludge	U-238	1.01E+01	1.48E+00	pCi/g	05/29/01	05/03/01	0.4046	82.0	A18	4.11E-01
1RD007033A	0105041-33	Sludge	U-234	3.86E+02	3.16E+01	pCi/g	05/29/01	05/03/01	0.4004	75.4	A19	4.46E-01
1RD007033A	0105041-33	Sludge	U-235	1.88E+01	2.31E+00	pCi/g	05/29/01	05/03/01	0.4004	75.4	A19	4.46E-01
1RD007033A	0105041-33	Sludge	U-238	5.59E+00	1.07E+00	pCi/g	05/29/01	05/03/01	0.4004	75.4	A19	4.46E-01
1RD007043A	0105041-34	Sludge	U-234	5.08E+02	4.16E+01	pCi/g	05/29/01	05/03/01	0.4043	76.2	A20	6.30E-01
1RD007043A	0105041-34	Sludge	U-235	2.79E+01	3.16E+00	pCi/g	05/29/01	05/03/01	0.4043	76.2	A20	5.86E-01
1RD007043A	0105041-34	Sludge	U-238	1.03E+01	1.60E+00	pCi/g	05/29/01	05/03/01	0.4043	76.2	A20	5.86E-01
1RD007053A	0105041-35	Sludge	U-234	4.48E+02	3.68E+01	pCi/g	05/29/01	05/03/01	0.4036	76.0	A22	5.57E-01
1RD007053A	0105041-35	Sludge	U-235	2.39E+01	2.84E+00	pCi/g	05/29/01	05/03/01	0.4036	76.0	A22	4.99E-01
1RD007053A	0105041-35	Sludge	U-238	7.82E+00	1.37E+00	pCi/g	05/29/01	05/03/01	0.4036	76.0	A22	4.99E-01
1RD008013A	0105041-36	Sludge	U-234	4.11E+03	3.35E+02	pCi/g	05/29/01	05/03/01	0.0408	81.2	A23	4.28E+00
1RD008013A	0105041-36	Sludge	U-235	1.73E+02	2.16E+01	pCi/g	05/29/01	05/03/01	0.0408	81.2	A23	4.28E+00
1RD008013A	0105041-36	Sludge	U-238	2.50E+01	6.76E+00	pCi/g	05/29/01	05/03/01	0.0408	81.2	A23	4.28E+00
1RD008023A	0105041-37	Sludge	U-234	4.06E+03	3.30E+02	pCi/g	05/29/01	05/03/01	0.0453	78.2	A24	3.98E+00
1RD008023A	0105041-37	Sludge	U-235	1.69E+02	2.07E+01	pCi/g	05/29/01	05/03/01	0.0453	78.2	A24	3.98E+00
1RD008023A	0105041-37	Sludge	U-238	1.95E+01	5.58E+00	pCi/g	05/29/01	05/03/01	0.0453	78.2	A24	3.98E+00
1RD008033A	0105041-38	Sludge	U-234	4.45E+03	3.67E+02	pCi/g	05/29/01	05/03/01	0.0452	80.6	A03	5.97E+00
1RD008033A	0105041-38	Sludge	U-235	1.41E+02	1.96E+01	pCi/g	05/29/01	05/03/01	0.0452	80.6	A03	4.90E+00
1RD008033A	0105041-38	Sludge	U-238	1.64E+01	5.64E+00	pCi/g	05/29/01	05/03/01	0.0452	80.6	A03	4.90E+00

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FORM I

## BBWI SAMPLE MANAGEMENT OFFICE

## RADIONUCLIDE ANALYSIS RESULTS

Date: May 29, 2001

Lab Name:

BWLVA

Case No.:

ER-SOW-380

Report No.:

0105041

SDG No.:

1RD001013A

BBWI ID	Lab ID	Sample Matrix	Anal Type	Sample Value	Sample Uncer	Units	Anal Date	Sample Date	Sample Size	Yield	Det ID	MDA
1RD008043A	0105041-39	Sludge	U-234	3.72E+03	3.06E+02	pCi/g	05/29/01	05/03/01	0.0408	85.0	A04	6.55E+00
1RD008043A	0105041-39	Sludge	U-235	1.65E+02	2.16E+01	pCi/g	05/29/01	05/03/01	0.0408	85.0	A04	7.27E+00
1RD008043A	0105041-39	Sludge	U-238	1.87E+01	6.55E+00	pCi/g	05/29/01	05/03/01	0.0408	85.0	A04	7.60E+00
1RD008053A	0105041-40	Sludge	U-234	4.64E+03	3.78E+02	pCi/g	05/29/01	05/03/01	0.0440	81.4	A05	5.56E+00
1RD008053A	0105041-40	Sludge	U-235	1.90E+02	2.30E+01	pCi/g	05/29/01	05/03/01	0.0440	81.4	A05	4.24E+00
1RD008053A	0105041-40	Sludge	U-238	2.95E+01	7.34E+00	pCi/g	05/29/01	05/03/01	0.0440	81.4	A05	4.24E+00
1RD009013A	0105041-41	Sludge	U-234	2.75E+03	2.29E+02	pCi/g	05/29/01	05/07/01	0.0432	80.8	A06	6.77E+00
1RD009013A	0105041-41	Sludge	U-235	1.07E+02	1.57E+01	pCi/g	05/29/01	05/07/01	0.0432	80.8	A06	4.37E+00
1RD009013A	0105041-41	Sludge	U-238	1.22E+01	4.77E+00	pCi/g	05/29/01	05/07/01	0.0432	80.8	A06	4.37E+00
1RD009023A	0105041-42	Sludge	U-234	3.34E+03	2.75E+02	pCi/g	05/29/01	05/07/01	0.0432	72.0	A07	9.24E+00
1RD009023A	0105041-42	Sludge	U-235	1.34E+02	1.84E+01	pCi/g	05/29/01	05/07/01	0.0432	72.0	A07	6.18E+00
1RD009023A	0105041-42	Sludge	U-238	1.11E+01	4.60E+00	pCi/g	05/29/01	05/07/01	0.0432	72.0	A07	4.43E+00
1RD009033A	0105041-43	Sludge	U-234	3.98E+03	3.27E+02	pCi/g	05/29/01	05/07/01	0.0467	88.6	A08	5.72E+00
1RD009033A	0105041-43	Sludge	U-235	1.39E+02	1.83E+01	pCi/g	05/29/01	05/07/01	0.0467	88.6	A08	4.10E+00
1RD009033A	0105041-43	Sludge	U-238	1.48E+01	5.08E+00	pCi/g	05/29/01	05/07/01	0.0467	88.6	A08	4.10E+00
1RD009043A	0105041-44	Sludge	U-234	3.36E+03	2.74E+02	pCi/g	05/29/01	05/07/01	0.0401	80.8	A18	5.48E+00
1RD009043A	0105041-44	Sludge	U-235	9.89E+01	1.37E+01	pCi/g	05/29/01	05/07/01	0.0401	80.8	A18	4.11E+00
1RD009043A	0105041-44	Sludge	U-238	1.38E+01	4.43E+00	pCi/g	05/29/01	05/07/01	0.0401	80.8	A18	3.37E+00
1RD009053A	0105041-45	Sludge	U-234	3.73E+03	3.04E+02	pCi/g	05/29/01	05/07/01	0.0423	80.2	A19	3.97E+00
1RD009053A	0105041-45	Sludge	U-235	1.26E+02	1.69E+01	pCi/g	05/29/01	05/07/01	0.0423	80.2	A19	3.97E+00
1RD009053A	0105041-45	Sludge	U-238	1.14E+01	4.40E+00	pCi/g	05/29/01	05/07/01	0.0423	80.2	A19	3.97E+00

Comments:

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## 1. INTRODUCTION

Nine samples were collected, each sample divided into five subparts by the laboratory (for a total of forty-five samples), and analyzed for Uranium-234/235/238 activity to support the Tank V-9 (TSF-18) Sampling for Operable Unit 1-10 project in support of Statement of Work (TOS) ER-SOW-380. The laboratory data package met the requested Level-A reporting requirements as per ER-SOW-163. The radioanalytical data were validated to analytical method data validation Level-A, in accordance with INEEL data validation procedures ~~TPR-79~~ & TPR-80 (References A & C).

GDE-7003

## 2. TASK SPECIFIC VALIDATION IDENTIFICATION INFORMATION

A.	L&V Report Number:	<u>BBWI-PR025-06-01</u>	F.	Reporting Level:	<u>Tier I</u>
B.	SDG Number:	<u>1RD001013A</u>	G.	Validation Level:	<u>A</u>
C.	Number of Samples:	<u>45</u>	H.	TOS Number:	<u>ER-SOW-380</u>
D.	Sample Type/Matrix:	<u>45 Sludge</u>	I.	Analytical Lab:	<u>BWLVA</u>
E.	Analysis Type:	<u>U-234/235/238</u>	J.	LTI Number:	<u>0105041</u>
			K.	Validator:	<u>Bridget Hoope</u>
			L.	Validator Affiliation:	<u>Portage</u>
			M.	Completion Date:	<u>06-12-01</u>

## 3. DATA VALIDATION PRECAUTIONS AND LIMITATIONS

It should be noted that this TPR describes method validation only and is not intended to provide guidance for validation of overall program/project objectives and requirements. Project validation is generally performed by project management personnel and involves a comprehensive review of all aspects (and objectives) of a sampling and analysis project.

The entire radioanalytical measurement process is a very elaborate process because it is composed of many elements and occurs in various phases/steps (from purchase, setup, calibration, and maintenance of detection systems, chemical separations/sample preparation processes, sample counting, analyses, reporting, and performance-monitoring of each of these elements). A considerable amount of information, data, and knowledge is generally required to technically support the accuracy, precision, and defensibility of each radioanalytical result. Enormous amounts of information and data are available at the laboratories that would probably be necessary in order to properly defend each radioanalytical result; however, it would be unreasonable to request all such data be included in each data package. It is the attempt of this procedure to achieve the best possible assurance of data defensibility and usability with the information available (required/requested) with each data package.

4. SAMPLE IDENTIFICATION

FIELD SAMPLE IDENTIFICATION NUMBER:	LABORATORY IDENTIFICATION NUMBER:	SAMPLE MATRIX:
1RD001013A	010541-01	Sludge
1RD001023A	010541-02	Sludge
1RD001033A	010541-03	Sludge
1RD001043A	010541-04	Sludge
1RD001053A	010541-05	Sludge
1RD002013A	010541-06	Sludge
1RD002023A	010541-07	Sludge
1RD002033A	010541-08	Sludge
1RD002043A	010541-09	Sludge
1RD002053A	010541-10	Sludge
1RD003013A	010541-11	Sludge
1RD003023A	010541-12	Sludge
1RD003033A	010541-13	Sludge
1RD003043A	010541-14	Sludge
1RD003053A	010541-15	Sludge
1RD004013A	010541-16	Sludge
1RD004023A	010541-17	Sludge
1RD004033A	010541-18	Sludge
1RD004043A	010541-19	Sludge
1RD004053A	010541-20	Sludge
1RD005013A	010541-21	Sludge
1RD005023A	010541-22	Sludge
1RD005033A	010541-23	Sludge

FIELD SAMPLE IDENTIFICATION NUMBER:	LABORATORY IDENTIFICATION NUMBER:	SAMPLE MATRIX:
1RD005043A	010541-24	Sludge
1RD005053A	010541-25	Sludge
1RD006013A	010541-26	Sludge
1RD006023A	010541-27	Sludge
1RD006033A	010541-28	Sludge
1RD006043A	010541-29	Sludge
1RD006053A	010541-30	Sludge
1RD007013A	010541-31	Sludge
1RD007023A	010541-32	Sludge
1RD007033A	010541-33	Sludge
1RD007043A	010541-34	Sludge
1RD007053A	010541-35	Sludge
1RD008013A	010541-36	Sludge
1RD008023A	010541-37	Sludge
1RD008033A	010541-38	Sludge
1RD008043A	010541-39	Sludge
1RD008053A	010541-40	Sludge
1RD009013A	010541-41	Sludge
1RD009023A	010541-42	Sludge
1RD009033A	010541-43	Sludge
1RD009043A	010541-44	Sludge
1RD009053A	010541-45	Sludge

## 5. CONTRACT AND TECHNICAL REVIEW (CTR)

This section contains the contract and technical review comments describing the findings and observations for each of the main verification and validation parameters described in TPR-80. The actions taken for each analysis and the reasons why a particular data qualifier flag was assigned are also included. The following verification and validation parameters were reviewed.

### A. COMPLETENESS OF THE DATA REPORT PACKAGE

The BWLVA data package bearing SDG#: 1RD001013A, was complete and met all the required Tier I reporting requirements described in ER-SOW-163 necessary to perform Level A data validation in accordance with TPR-80.

### B. EVALUATION OF REPORTED RESULTS

For level-A data validation, evaluation of reported results versus raw data is applicable. All supporting materials provided indicate results were reported in accordance with the requirements set forth in ER-SOW-163.

All of the sample-specific information for each sample has been reported correctly. Sample results obtained from spectrometric analysis do not require verification when the results were obtained from computer analysis software that has received approval by the INEEL Sample Management office.

Per TPR-80, sec. 3.2.C.11, a minimum of 10% of analytical results have been checked to verify that the calculations were performed correctly and consistently; all reported results that were verified versus their associated raw data demonstrated that reported results are accurate.

### C. DETECTOR SYSTEM CALIBRATIONS AND OPERATIONAL PERFORMANCE CHECKS

All calibrations, calibration verification checks, and background checks provided support the "In control" designation reported on each of the ER-SOW-163 Form IIIs. Therefore, the detector calibrations were in control and the instruments were operating properly during the counting/analysis of the reported sample results; no qualification is warranted.

### D. LABORATORY CONTROL SAMPLE RESULTS

The laboratory control sample (LCS) is analyzed to demonstrate that the recovery of the requested nuclide of interest is accurate; the acceptable or out of compliance performance of the LCS directly reflects on the effectiveness of the analytical process from sample preparation through instrumental measurements.



TPR-80, sec. 4.2.B.2 requires "the LCS matrix should be equivalent (to the extent possible) to that of the samples analyzed." The samples included in this SDG are of a sludge matrix, while the LCS analyzed is of a liquid matrix. Due to the difficulty of preparing a "sludge" LCS, the use a liquid LCS is equivalent to the extent possible of a "sludge" LCS.

LCS recoveries were provided for each of the isotopes of interest. All LCS results provided have met the limits of 70-130% recovery for gross alpha and gross beta measurements, with the exception of U-235, outlined in TPR-80, sec. 4.2.C.4.

In the case of U-235 (137.7%), one of the three LCS results was greater than the prescribed limit. Therefore, per TPR-80, sec. 4.2.D.3, all U-235 results associated with preparation batch 511-41, as noted in the raw data, have been qualified 'J' due to high LCS recovery and statistically positive results greater than the MDA. Preparation batch 511-41 is composed of samples 1RD001013A, 1RD001023A, 1RD001033A, 1RD001043A, 1RD001053A, 1RD002013A, 1RD002023A, 1RD002033A, 1RD002043A, 1RD002053A, 1RD003013A, 1RD003023A, 1RD003033A, 1RD003043A, and 1RD003053A.

#### E. LABORATORY METHOD BLANK RESULTS

A laboratory generated blank sample (or method blank) analyzed for each sample delivery group (1-blank : 20 samples) is a means of determining the existence and magnitude of contamination resulting from the sample preparation and analysis/measurement process. Any statistically positive activity detected for a target radionuclide indicates a potential positive bias in the project sample results associated with statistically positive nuclides.

Three laboratory generated blanks were analyzed with this SDG for each applicable target radionuclide. There were no statistically positive results noted for any target radionuclides. Therefore, no qualification is necessary per TPR-80, sec. 4.3.D.1.a.

#### F. LABORATORY GENERATED DUPLICATE RESULTS

The information obtained from the analysis of laboratory generated duplicates is useful to evaluate analytical variability and laboratory precision. Results from the analysis of laboratory generated duplicate samples can also reflect the homogeneity or inhomogeneity of individual samples or groups of samples of the same matrices. For a duplicate sample to meet the acceptance criteria outlined in TPR-80 - Section 4.4, sample precision must be  $\leq 3$  for the mean difference (MD) and/or  $\leq 20\%$  relative percent difference (RPD) for water samples. However, the mean difference takes precedence over the calculation and use of RPD for duplicate precision (TPR-80 - Section 4.4, Subsection 2).

Duplicate sample results for all isotopes demonstrated acceptable laboratory precision with MD values  $< 3$ ; all results exhibited statistically positive sample results (See Attachment 5). Per TPR-80, sec. 4.4.C.4, the RPD calculation does not need to be calculated when the MD value is  $< 3$ .

G. LABORATORY ANALYTICAL YIELDS

The evaluation of an analytical yield serves to evaluate the efficiency of radiochemical separations utilized when preparing samples for measurement or analysis. The use of a tracer is conducted when a known amount of a chemical tracer is added to unknown samples; during analysis, a yield or recovery of the tracer material is used to determine the efficiency of the entire analytical process. The tracer that is chosen is used because it mimics the properties of one or more target radionuclides.

The sample analysis of U-234/235/238 met the tracer recovery criteria of 30-110% outlined for uranium analysis of natural and QC samples, per TPR-80, sec. 4.5.C.

H. ANALYTICAL HOLDING TIMES

The holding time requirements (i.e.  $< 6$  months) were met for this SDG.

I. FIELD SAMPLE PRESERVATION

None of the samples associated with this SDG were of a liquid nature; therefore, they did not require preservation.

J. LABORATORY INTERCOMPARISON QC RESULTS

The Intercomparison QC testing program currently includes participation in the following QC programs: The U.S. Department of Energy (DOE) Environmental Measurement Laboratory (EML) Quality Assessment Program (QAP) and the U.S. DOE Office of Environmental Management, Mixed Analyte Performance Evaluation Program (MAPEP). Although, laboratory intercomparison QC results were not provided for DOE EML QAP, results for DOE MAPEP were provided for all isotopes from each analysis type applicable to this SDG.

BWLVA received a warning flag ('W') from the DOE MAPEP intercomparison conducted in 2000 due to high bias in analysis of U-233/234. This deficiency, coupled with reporting only DOE MAPEP results has resulted in the entry of a 'Q' flag into the data quality assessment table for each analyte. However, per TPR-80, sec. 4.8.A(NOTE), and TPR-80, sec. 4.8.D.2, no qualifier flags have been assigned to sample results.

K. INEEL PE SAMPLE RESULTS

There were no INEEL performance evaluation samples noted in the transmittal of this report, nor on any of the official documents. Therefore, no evaluation of INEEL PE standards was conducted.

6. **DATA LIMITATIONS AND USABILITY OVERVIEW**

This section provides an overview of the limitations of the data for each sample and for each analysis.

6.1 **Summary of Qualified Data**

All samples exhibited positive detections of radioisotope activity in the samples associated with the Tank V-9 (TSF-18) Sampling for Operable Unit 1-10 Project. Statistically positive sample results greater than their respective MDA's are listed in Table 6.0.

Table 6.0. Summary of Statistically Positive Results by Sample	
1RD001013A	U-234, U-235, U-238
1RD001023A	U-234, U-235, U-238
1RD001033A	U-234, U-235, U-238
1RD001043A	U-234, U-235, U-238*
1RD001053A	U-234, U-235, U-238
1RD002013A	U-234, U-235, U-238
1RD002023A	U-234, U-235, U-238
1RD002033A	U-234, U-235, U-238
1RD002043A	U-234, U-235, U-238
1RD002053A	U-234, U-235, U-238
1RD003013A	U-234, U-235, U-238
1RD003023A	U-234, U-235, U-238
1RD003033A	U-234, U-235, U-238
1RD003043A	U-234, U-235, U-238
1RD003053A	U-234, U-235, U-238
1RD004013A	U-234, U-235, U-238
1RD004023A	U-234, U-235, U-238

Table 6.0. Summary of Statistically Positive Results by Sample	
1RD004033A	U-234, U-235, U-238
1RD004043A	U-234, U-235, U-238
1RD004053A	U-234, U-235, U-238
1RD005013A	U-234, U-235, U-238
1RD005023A	U-234, U-235, U-238
1RD005033A	U-234, U-235, U-238
1RD005043A	U-234, U-235, U-238
1RD005053A	U-234, U-235, U-238
1RD006013A	U-234, U-235, U-238
1RD006023A	U-234, U-235, U-238
1RD006033A	U-234, U-235, U-238
1RD006043A	U-234, U-235, U-238
1RD006053A	U-234, U-235, U-238
1RD007013A	U-234, U-235, U-238
1RD007023A	U-234, U-235, U-238
1RD007033A	U-234, U-235, U-238
1RD007043A	U-234, U-235, U-238
1RD007053A	U-234, U-235, U-238
1RD008013A	U-234, U-235, U-238
1RD008023A	U-234, U-235, U-238
1RD008033A	U-234, U-235, U-238*
1RD008043A	U-234, U-235, U-238*
1RD008053A	U-234, U-235, U-238
1RD009013A	U-234, U-235, U-238*
1RD009023A	U-234, U-235, U-238*
1RD009033A	U-234, U-235, U-238*
1RD009043A	U-234, U-235, U-238
1RD009053A	U-234, U-235, U-238*

\*Per guidance provided by the INEEL SMO these sample results may be interpreted as statistically positive as follows:

The U-238 results for samples 1RD001043A, 1RD008033A, 1RD008043A, 1RD009013A, 1RD009023A, 1RD009033A, and 1RD009053A have been qualified 'J' due to activity that is greater than the MDA but between 2x and 3x the uncertainty associated with the result.

Remaining sample results are summarized below.

Uranium-234/235/238 Analyses

The U-234/235/238 sample results for all samples, except the U-238 results for samples 1RD001043A, 1RD008033A, 1RD008043A, 1RD009013A, 1RD009023A, 1RD009033A, and 1RD009053A, demonstrated statistically positive activities greater than their respective MDAs and greater than 3x their respective uncertainties. Therefore, no validator action was necessary on these sample results.

U-235 results associated with preparation batch 511-41 (composed of samples 1RD001013A, 1RD001023A, 1RD001033A, 1RD001043A, 1RD001053A, 1RD002013A, 1RD002023A, 1RD002033A, 1RD002043A, 1RD002053A, 1RD003013A, 1RD003023A, 1RD003033A, 1RD003043A, and 1RD003053A) have been qualified 'J' due to a high LCS result (137.7% recovery) and statistically positive results greater than their respective MDAs.

Determination of the statistically positive or not statistically positive status of sample results is provided in Attachment 6, Supplemental Validation - TPR-80.

The laboratory case narrative notes that "several alpha spectra showed breakthrough from the high concentration of plutonium isotopes inherent in the samples, however, the uranium peaks were easily resolved." Because the uranium peaks were resolved from the plutonium breakthrough, qualification of results is not necessary.

## 6.2 Radioanalytical Data Quality Assessment Table

Project Name: Tank V-9 (TSF-18) Sampling for Operable Unit 1-10 Project

L&V Report#: BBWI-PR025-06-01 Validation Level: A Assessor's Affiliation: Portage  
SDG#: 1RD001013A Reporting Level: Tier I Assessor's Name: Bridget Hoopes  
TOS#: ER-SOW-380 Samples by Matrix: 45 Sludge Assessment Date: 06-12-01  
SOW#: ER-SOW-163 Laboratory Name: BWLVA

	Analysis Type:			Comment	
	U-234	U-235	U-238	Y	N
<b>Verification Review Parameters</b>					
1. Data Package Completeness	I	I	I		X
2. Evaluation of Reported Results	I	I	I		X
<b>Validation Review Parameters</b>					
1. Instrument Calibrations	I	I	I		X
2. Laboratory Control Sample	I	Q	I	X	
3. Blank Samples	I	I	I		X
4. Duplicate Samples	I	I	I		X
5. Analytical Yields	I	I	I		X
6. Sample Holding Times	I	I	I		X
7. Sample Preservation	NA	NA	NA		X
8. Intercomparison QC Results	Q	Q	Q	X	
9. INEEL PE Sample Results	NA	NA	NA		X

### Quality Assurance Flags:

- I Parameter is in control (meets acceptance criteria). There are no problems with the sample results
- Q Parameter is questionable. There may be minor problems with the sample results data.
- O Parameter is out of control (does not meet acceptance criteria). There may be major problems with the sample results data.
- NA Parameter is not acceptable to this analysis.
- Y Yes indicates a comment was made and be found on the comment sheet.
- N No indicates no comment was made.

### 6.3 Radioanalytical Data Quality Assessment Sheet

Project Name: Tank V-9 (TSF-18) Sampling for Operable Unit 1-10 Project

L&V Report#: BBWI-PR025-06-01

Laboratory Name: BWLVA

Date: 06-12-01

REVIEW PARAMETER	COMMENT
1. <i>Laboratory Control Sample</i>	<p>TPR-80, sec. 4.2.B.2 requires "the LCS matrix should be equivalent (to the extent possible) to that of the samples analyzed." The samples included in this SDG are of a sludge matrix. The LCS analyzed is of a liquid matrix. Due to the difficulty of preparing a "sludge" LCS, the use a liquid LCS is equivalent to the extent possible of a "sludge" LCS.</p> <p>In the case of U-235, one of the three LCS results was greater than the prescribed limit (137.7%). Therefore, per TPR-80, sec. 4.2.D.3, all U-235 results associated with preparation batch 511-41 (composed of samples 1RD001013A, 1RD001023A, 1RD001033A, 1RD001043A, 1RD001053A, 1RD002013A, 1RD002023A, 1RD002033A, 1RD002043A, 1RD002053A, 1RD003013A, 1RD003023A, 1RD003033A, 1RD003043A, and 1RD003053A) have been qualified 'J' due to high LCS recovery and statistically positive results greater than their MDAs.</p>
2. <i>Intercomparison QC Results</i>	<p>BWLVA received a warning flag ('W') from the DOE MAPEP intercomparison conducted in 2000 due to high bias for U-233/234 analysis. This deficiency, coupled with reporting only DOE MAPEP results has resulted in the entry of a 'Q' flag into the data quality assessment table for each analyte. However, per TPR-80, sec. 4.8.A(NOTE), and TPR-80, sec. 4.8.D.2, no qualifier flags have been assigned to sample results.</p>

## 6.4 Radioanalytical Data Qualifier (Validation Flag) Table

Project Name: Tank V-9 (TSF-18) Sampling for Operable Unit 1-10 Project

L&amp;V Report#: BBW1-PR025-06-01

SDG#: IRD001013A

TOS#: ER-SOW-380

SOW#: ER-SOW-163

Validation Level: A

Reporting Level: Tier I

Samples by Matrix: 45 Sludge

Laboratory Name: BWLVA

Assessor's Affiliation: Portage Environmental, Inc.

Assessor's Name: Bridget Hoopes

Assessment Date: 06-12-01

SAMPLE NUMBER	ANALYSIS TYPE			SAMPLE NUMBER	ANALYSIS TYPE		
	U-234	U-235	U-238		U-234	U-235	U-238
IRD001013A		J		IRD004033A			U-238
IRD001023A		J		IRD005013A			
IRD001033A		J		IRD005023A			
IRD001043A		J	J	IRD005033A			
IRD001053A		J		IRD005043A			
IRD002013A		J		IRD005053A			
IRD002023A		J		IRD006013A			
IRD002033A		J		IRD006023A			
IRD002043A		J		IRD006033A			
IRD002053A		J		IRD006043A			
IRD003013A		J		IRD006053A			
IRD003023A		J		IRD007013A			
IRD003033A		J		IRD007023A			
IRD003043A		J		IRD007033A			
IRD003053A		J		IRD007043A			
IRD004013A				IRD007053A			
IRD004023A				IRD008013A			
IRD004033A				IRD008023A			
IRD004043A				IRD008033A			J



Table 6.4, Continued...

SAMPLE NUMBER:	ANALYSIS TYPE:		
	U-234	U-235	U-238
1RD008043A			J
1RD008053A			
1RD009013A			J
1RD009023A			J
1RD009033A			J
1RD009043A			
1RD009053A			J

- none The analysis was performed and radioactivity was detected (e.g., the radioanalytical result is statistically positive at the 95% confidence level and is above the MDC). The radionuclide is considered to be present in the sample.
- N/P This analysis was not a requirement of this analytical request for the marked sample.
- U The analysis was performed, but no radioactivity was detected (i.e., the radioanalytical result was not statistically positive at the 95% confidence level and/or the result was below the MDC). NOTE: *The radionuclide is not considered to be present in the sample.*
- UJ The analysis was performed and the result is highly questionable due to serious analytical and/or laboratory quality control anomalies. The use of such a result is strongly discouraged. Serious analytical and/or quality control anomalies include items such as significant blank contamination, known photopeak interferences, or photopeak resolution problems, known matrix interferences, unacceptable laboratory control sample recoveries, serious instrument calibration problems, improper sample preservation, etc. NOTE: *The radionuclide may or may not be present and the result is considered highly questionable.*
- J The analysis was performed and radioactivity was detected (i.e., the radioanalytical result is statistically positive at the 95% confidence level and is above the MDC). However, the result is questionable due to analytical and/or laboratory quality control anomalies and should therefore be used only as an estimated (approximated) quantity. Analytical and/or quality control anomalies include items such as: laboratory duplicate imprecision, unsatisfactory analytical yields, insufficient laboratory control sample recoveries, unacceptable PE sample results, instrument calibration problems, improper sample preservation, etc. NOTE: *The radionuclide is considered to be present, but the result may inaccurate or imprecise.*
- R The analysis result is unusable and was rejected due to severe analytical and/or quality control problems. NOTE: *The radionuclide may or may not be present and the result is known to be inaccurate or imprecise.*

#### 6.5 Summary of Data Usability

There were 135 radionuclide results associated with the Tank V-9 (TSF-18) Sampling for Operable Unit 1-10 Project in SDG: 1RD001013A. One hundred thirteen (113) of these results were assessed and left unqualified, and twenty-two (22) of these results were assessed and qualified 'J'.

All target radionuclides demonstrated compliance with the requirements specified in ER-SOW-163, ER-SOW-380, and TPR-80. Of the (135) total results reported, the (113) assessed and left unqualified and the (7) U-238 results qualified 'J' can be categorized as definitive/useable data with no associated quality assessment deficiencies. The remaining (15) U-235 results have been assessed and qualified 'J' because one of three laboratory control samples exceeded recovery limits, which may indicate high biased sample results. All (113) unqualified samples, as well as all (15) U-235 sample results are statistically positive with activities both greater than their respective MDAs and greater than their respective uncertainties. The (7) U-238 results qualified 'J' are statistically positive with activities greater than their associated MDAs and between 2x and 3x their associated uncertainty.

#### 7. FLAGRANT CONTRACTUAL DEFICIENCIES

None.

## **TCLP Metal Analysis Data and Data Validation**



**TCLP INORGANIC ANALYSIS DATA SHEET**

Contract 00000194, R1

SDG# IRD001013A

Lab ID# 0105041-46AA

Concentration Units: ug/L

[illegible]

"F" - Furnace  
 "P" - ICP  
 "CV" - Cold vapor

Comments:

SDG TYPE-2 FORM #1

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**TCLP INORGANIC ANALYSIS DATA SHEET**

Contract 00000194, R1

SDG# IRD001013A

Lab ID# 0105041-47AA

Concentration Units: ug/L

[illegible]

"F" - Furnace  
 "P" - ICP  
 "CV" - Cold vapor

Comments:

SDG TYPE-2 FORM #1

000022

1.0 Task Specific Validation Identification Information

1. L&V Report Number: BBWI-PI336-06-01
2. SDG Number: 1RD001013A
3. SDG Type: 2
4. Number of Samples: (2)
5. Sample Matrix: (2) Sludge (TCLP Extracted)
6. Applicable Analytes: Toxicity Characteristic Leaching Procedure (TCLP)  
Target Analyte List (TAL): (As, Ba, Cd, Cr, Pb, Hg, Se, and Ag)
7. Reporting Tier: Tier-1
8. Applicable TOS#: ER-SOW-380
9. TOS Title: *Analyses of Samples Collected for the Tank V-9 (TSF-18) Sampling for Operable Unit 1-10 Project; ER-SOW-380; April 23, 2001*
10. Analytical Lab: BWXT
11. LTI Number: 0105041
12. Validator: Jennifer Norman
13. Validator Affiliation: Portage Environmental, Inc.
14. Validation Level: 'A'
15. Completion date: 06-14-01

## 2.0 IDV Precautions and Limitations

General precautions and limitations associated with inorganic and miscellaneous classical analyses analytical method data validation (IDV) are delineated in Section 2 of TPR-132 (*Reference 1*).

## 3.0 Introduction

Level 'A' inorganic data validation (IDV) [see TPR-79 (*Reference 2*)], following the procedures outlined in TPR-132 (*Reference 1*), was performed on the inorganic data package (IDP), identified as sample delivery group (SDG) number 1RD001013A, compiled by BWXT. TPR-132 is an Idaho National Engineering & Environmental Laboratory (INEEL) Sample Management Office (SMO) document that has revised the validation procedures outlined in the United States Environmental Protection Agency (USEPA) Functional Guidelines (*Reference 3*) to more aptly apply to IDPs prepared in accordance with the generic inorganic and miscellaneous classical analyses (I&MCA) statement of work [see ER-SOW-156 (*Reference 4*)] routinely requested by the INEEL SMO. BWXT analyzed (2) of (2) sludge TCLP extracted samples for As, Ba, Cd, Cr, Pb, Hg, Se, and Ag.

BWXT was contracted to analyze (2) of (2) sludge TCLP extracted samples for As, Ba, Cd, Cr, Pb, Hg, Se, and Ag; under this contract, they were to perform sample extraction in accordance with USEPA SW-846 Method 1311 (*Reference 5*), sample preparation and analysis of mercury in accordance with USEPA SW-846 Method 7470A (*Reference 6*), sample preparation of remaining analytes in accordance with USEPA SW-846 Method 3015A (*Reference 7*), and sample analysis in accordance with USEPA SW-846 Method 6010B (*Reference 8*) in conjunction with both the task order statement of work [see ER-SOW-380 (*Reference 9*)] and ER-SOW-156. The laboratory performed analysis of the ICP metals in accordance with USEPA SW-846 Method 6010A. This method is an earlier version of Method 6010B. Therefore, this method substitution is acceptable and warrants no qualification of sample results. The laboratory performed the analysis of the Tank V-9 Sampling for Operable Unit 1-10 sludge TCLP extracted samples using appropriate methods.

## 4.0 Sample Identification

The following table outlines the field sample identifiers, the laboratory identifications, and the appropriate sample matrix assigned to each analyte.

| Table 4.0 Sample Identifications for the Tank V-9 Sampling for Operable Unit 1-10<br>Sludge TCLP Extracted Samples |                 |                         |
|--------------------------------------------------------------------------------------------------------------------|-----------------|-------------------------|
| Field Sample Id#:                                                                                                  | Laboratory Id#: | Original Sample Matrix: |
| 1RD01001TI                                                                                                         | 0105041-46      | Sludge                  |
| 1RD01002TI                                                                                                         | 0105041-47      | Sludge                  |



## 5.0 Contract and Technical Review

1. The laboratory case narrative contained all of the elements outlined in ER-SOW-156.
2. The laboratory holding time critique and chain of custody forms were complete and accurate.
3. All analytes were analyzed within the 28-day holding time for mercury and the 180-day holding time for all remaining analytes as prescribed by ER-SOW-380.
4. All AQS calibration results demonstrated a correlation coefficient greater than 0.995 as prescribed by TPR-132, sec. 4.3.2.4.
5. All initial calibration verification (ICV) sample results were within the 90-110% recovery criteria as prescribed by TPR-132, sec. 4.3.3.5.5.
6. Barium (89.4%) was outside the 90-110% recovery criteria for continuing calibration verification sample results, per TPR-132, sec. 4.3.4.5.5. All barium sample results have been qualified with a 'J' validation flag due to sample results that are greater than the IDL and low CCV recovery. All remaining CCV sample results were within the 90-110% recovery criteria as outlined in TPR-132, sec. 4.3.4.5.5.
7. Low level concentration sample results were within the 50-150% acceptance criteria outlined in TPR-132, sec. 4.3.5.5.5.
8. Initial calibration blank results were all non-detect, per TPR-132, sec. 4.3.6.5.5. A positive detection for selenium was noted in the continuing calibration blank. Positive detections were noted in the preparation blank for arsenic, barium, and selenium. All remaining ICB, CCB, and PB results were all non-detect and do not warrant qualification, per TPR-132, sec. 4.3.6.5.5 and 4.3.7.5.5. In the case of multiple detections, assessment is based on the highest absolute value detection as follows:
  - Arsenic results have been assessed using positive PB criteria. Arsenic sample result 1RD01001TI does not warrant qualification as the sample result is less than the IDL, per TPR-132, sec. 4.3.7.5.5. Arsenic sample result 1RD01002TI has been qualified with a 'U' validation flag due to the sample result being greater than the IDL but less than five times the amount of analyte found in the blank, per TPR-132, sec. 4.3.7.5.5.
  - Barium results have been assessed using positive PB criteria. All barium sample results do not warrant qualification as the sample results are greater than the IDL and greater than five times the amount of analyte found in the blank, per TPR-132, sec. 4.3.7.5.5.
  - Selenium results have been assessed using positive PB criteria. Selenium sample result 1RD01001TI does not warrant qualification as the sample results is less than the IDL, per TPR-132, sec. 4.3.7.5.5. Selenium sample result 1RD01002TI has been qualified with a 'U' validation flag due to the sample result being greater than the IDL but less than five times the amount of analyte found in the blank, per TPR-132, sec. 4.3.7.5.5.
9. ICP-ICS results were within the 80-120% acceptance criteria as outlined in TPR-132, sec. 4.3.8.5.

10. Barium (78.5%), cadmium (77.8%), and silver (66.4%) were outside the 80-120% recovery criteria for matrix spike (MS) results. The laboratory failed to 'N' flag barium and cadmium results; the 'N' flag for these results has been entered during validation.

- Barium results would warrant qualification with a 'J' flag; however, barium has already been qualified based upon CCV results. This adds further merit to the 'J' qualification of barium results based upon CCV recovery results.
- Cadmium results have been qualified with a 'J' validation flag due to low percent recovery and sample results that are greater than the IDL, per TPR-132, sec. 4.3.9.5.5.
- Silver results have been qualified with a 'UJ' validation flag due to low percent recovery and sample results that are less than the IDL, per TPR-132, sec. 4.3.9.5.5.

Barium (79.0%), cadmium (78.6%), and silver (73.4%) were outside the 80-120% recovery criteria for matrix spike duplicate results. The laboratory failed to 'N' flag barium and cadmium results; the 'N' flag for these results has been entered during validation.

- Barium results would warrant qualification with a 'J' validation flag; however, barium has already been qualified based upon CCV and MS recovery results. This adds further merit to the 'J' qualification of barium based upon CCV and MS recovery results.
- Cadmium results would warrant qualification with a 'J' validation flag; however, cadmium results have already been qualified based upon MS recovery results. This adds further merit to the 'J' qualification of cadmium results based upon MS recovery results.
- Silver results would warrant qualification with a 'UJ' validation flag; however, silver results have already been qualified based upon MS recovery results. This adds further merit to the 'UJ' qualification of silver based upon MS recovery results.

11. Cadmium (-94.8%) and silver (64.0%) were outside the 75-125% recovery criteria for analytical spikes (AS), per ERD-SOW-107R2, sec. 4.4.7. AS results are assessed in conjunction with MS and serial dilution sample (SDS) results, to determine whether or not method of standard additions (MSA) is warranted. The laboratory failed to 'E' flag cadmium and silver results. So, the validator manually entered the 'E' flag for cadmium and silver during validation. The low percent recovery of cadmium adds further merit to the 'J' qualification of results based upon MS and MSD recovery results. The low percent recovery of silver adds further merit to the 'UJ' qualification of results based upon MS and MSD recovery results. However, MSA is not warranted due to MS and MSD recovery being greater than 50% and sample concentration being within 20% of the appropriate regulatory level, per TPR-132, sec. 4.3.9.5.5 subpart 4 and sec. 4.3.10.5.5.
12. Matrix spike duplicate results met the precision criteria of an RPD of less than 20% per TPR-132, sec. 4.3.12.5.4.

13. All aqueous laboratory control sample results were within the 80-120% acceptance criteria as outlined in TPR-132, sec. 4.3.13.5.5.
14. Serial dilution sample (SDS) results are assessed in conjunction with MS and AS results to determine whether or not MSA is warranted. All serial dilution results met the acceptance criteria of a percent difference less than 10% for analytes whose concentrations are minimally fifty times greater than the IDL as prescribed in ER-SOW-156, sec. 3.6.14.4. Therefore, per TPR-132, sec. 4.3.15.5.5, MSA is not warranted.
15. Linear range analysis sample results were within the 95-105% acceptance criteria as prescribed by TPR-132, sec. 4.3.16.5.5.

## 6.0 Data Limitations Overview

### 6.1 Summary of Qualified Data

- Arsenic sample result 1RD01002TI has been qualified with a 'U' validation flag to denote that the data is non-detectable at the reported value due to positive PB detections (See CTR Comment #8).
- All barium sample results have been qualified with a 'J' validation flag to denote that the data is detectable at the reported value but that the reported value is only an estimate due to low CCV, MS, and MSD recovery (See CTR Comments #6 & #10).
- All cadmium sample results have been qualified with a 'J' validation flag to denote that the data is detectable at the reported value but that the reported value is only an estimate due to low MS, MSD, and AS recovery (See CTR Comments #10 & #11).
- Selenium sample result 1RD01002TI has been qualified with a 'U' validation flag to denote that the data is non-detectable at the reported value due to positive PB detections (See CTR Comment #8).
- All silver sample results have been qualified with a 'UJ' validation flag to denote that the data is non-detectable at the reported value but the reported value is only an estimate due to low MS, MSD, and AS recovery (See CTR Comments #10 & #11).
- All remaining data points have been assessed and remain unqualified.

## 6.2 Data Confirmation Summary

Table 6.2 includes summary of correctly/incorrectly reported results for SDG#: 1RD001013A.

| FIELD SAMPLE DATA POINTS ASSOCIATED WITH SDG#: 1RD001013A |                  |                                           |                                         |                                        |
|-----------------------------------------------------------|------------------|-------------------------------------------|-----------------------------------------|----------------------------------------|
| Total Number                                              | Number Confirmed | Number Confirmed to be Correctly Reported | Number Confirmed to be Falsely Reported | Actual Proportion Falsely Reported (%) |
| 16                                                        | 16               | 16                                        | 0                                       | 0                                      |

### 6.3 Data Assessment Table

| L&V Report#: | BBWI-PI336-06-01             | Validation Level: | 'A'                         | Assessor Affiliation: | Portage Environmental, Inc. |     |     |     |     |
|--------------|------------------------------|-------------------|-----------------------------|-----------------------|-----------------------------|-----|-----|-----|-----|
| SDG#:        | IRD001013A                   | SDG Type:         | 2                           | Assessor Name:        | Jennifer Norman             |     |     |     |     |
| TOS#:        | ER-SOW-380                   | Reporting Tier:   | 1                           | Assessor Signature:   | <i>Jennifer Norman</i>      |     |     |     |     |
| SOW#:        | ER-SOW-156                   | Samples/Matrix:   | (2) Sludge (TCLP extracted) | Assessment Date:      | 06-14-01                    |     |     |     |     |
| LTI#:        | 0105041                      | Laboratory:       | BWXT                        |                       |                             |     |     |     |     |
| Item#        | Assessment Item:             | Target Analyte    |                             |                       |                             |     |     |     |     |
|              |                              | As                | Ba                          | Cd                    | Cr                          | Pb  | Hg  | Se  | Ag  |
| 1            | Holding Times                | O                 | O                           | O                     | O                           | O   | O   | O   | O   |
| 2            | AQS Calibration              | O                 | O                           | O                     | O                           | O   | O   | O   | O   |
| 3            | ICV                          | O                 | O                           | O                     | O                           | O   | O   | O   | O   |
| 4            | CCV                          | O                 | M                           | O                     | O                           | O   | O   | O   | O   |
| 5            | LLC Standard (CRI in IDP)    | O                 | O                           | O                     | O                           | O   | O   | O   | O   |
| 6            | ICBs/CCBs                    | O                 | O                           | O                     | O                           | O   | O   | X   | O   |
| 7            | Preparation Blank            | X                 | X                           | O                     | O                           | O   | O   | X   | O   |
| 8            | ICP ICSA/ICSAB               | O                 | O                           | O                     | O                           | O   | O   | O   | O   |
| 9            | Matrix Spike                 | O                 | M                           | M                     | O                           | O   | O   | O   | M   |
| 10           | Analytical Spike             | O                 | O                           | X                     | O                           | O   | O   | O   | X   |
| 11           | Laboratory Duplicate         | N/A               | N/A                         | N/A                   | N/A                         | N/A | N/A | N/A | N/A |
| 12           | Matrix Spike Duplicate       | O                 | M                           | M                     | O                           | O   | O   | O   | M   |
| 13           | Lab/Method Control Sample    | O                 | O                           | O                     | O                           | O   | O   | O   | O   |
| 14           | Method of Standard Additions | N/A               | N/A                         | N/A                   | N/A                         | N/A | N/A | N/A | N/A |
| 15           | Serial Dilutions             | O                 | O                           | O                     | O                           | O   | O   | O   | O   |
| 16           | LRA Standard                 | O                 | O                           | O                     | O                           | O   | O   | O   | O   |
| 17           | CRC Standard                 | N/A               | N/A                         | N/A                   | N/A                         | N/A | N/A | N/A | N/A |
| 18           | Data Confirmation            | O                 | O                           | O                     | O                           | O   | O   | O   | O   |
| 19           | Miscellaneous Items          | O                 | O                           | O                     | O                           | O   | O   | O   | O   |
| 20           | Overall Assessment           | X                 | SI                          | M                     | O                           | O   | O   | X   | M   |

### 6.3 Data Assessment Table (cont...)

- O = Assessment item was in applicable control limits and, if considered alone, would not cause any data to be assigned a "UJ", "J," or "R" validation flags.
- M = Assessment item was outside applicable control limits and, if considered alone, would: (a) cause one or more field sample data points to be assigned either a "UJ" or "J" validation flag, but (b) not cause any data to be assigned a "R" validation flag.
- Z = Assessment item was outside applicable control limits and, if considered alone, would cause one or more field data points to be assigned a "R" validation flag.
- N/A = Assessment item not applicable.
- NP = Assessment item was required but was not performed and/or documented by the laboratory.
- X = Contractual and/or technical anomalies were noted but, based on the professional judgment of the assessor, none of the associated data were adversely affected.
- I = Contractual and/or technical anomalies were noted and, based on the professional judgement of the assessor, at least a portion of the data were adversely affected and/or could not be properly assessed. As a result, at least one applicable field sample data point was qualified with either a "UJ", "J", or "R" validation flag.
- G = The units reported for at least one applicable field sample data point did not correlate with the test method employed.

#### 6.4 Data Validation Flag Table

| Target Analyte and Assigned Qualification: SDG#: 1RD001013A |    |    |    |    |    |    |    |    |
|-------------------------------------------------------------|----|----|----|----|----|----|----|----|
| Field Sample Id#:                                           | As | Ba | Cd | Cr | Pb | Hg | Se | Ag |
| 1RD01001TI                                                  |    | J  | J  |    |    |    |    | UJ |
| 1RD01002TI                                                  | U  | J  | J  |    |    |    | U  | UJ |

#### Definitions of Data Validation Flags

- U - The material was analyzed for and was detected at or above the applicable detection limit. However, the associated value was less than 5 times the highest positive amount in any laboratory blank. In most instances the "U" validation flag will be accompanied by a "B" laboratory flag.
- UJ - The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise. A "UJ" validation flag is not differentiated from the combined action of both a "U" and "J" validation flag.
- J - The material was analyzed for and was detected at or above the applicable detection limit. The associated value is an estimate and may be inaccurate or imprecise.
- R - The accuracy of the data is so questionable that it is recommended the data not be used. For any given data point, a "R" validation flag overrides all other applicable flags.

#### 6.5 Summary of Data Usability

There were 16 field sample data points associated with the SDG# 1RD001013A IDP. Of these field sample data points: (a) 8 were assessed and left unqualified, (b) 2 were assessed and qualified with a 'U' validation flag, (c) 2 were assessed and qualified with a 'UJ' validation flag, and (d) 4 were assessed and qualified with a 'J' validation flag. Using the criteria outlined in EPA540-R-93-071 (*Reference 10*): (1) the 8 field sample data points (50% of the total) that were assessed and left unqualified can be categorized as definitive data with no associated quality control deficiencies, (2) the 2 field sample data points (12.5% of the total) that were assessed and qualified with a 'U' validation flag can be categorized as definitive data with a non-detectable analyte concentration due to positive blank detections, (3) the 2 field sample data points (12.5% of the total) that were assessed and qualified with a 'UJ' validation flag can be categorized as definitive data with a non-detectable analyte concentration that is only an estimate due to low MS, MSD, and AS recovery, and (4) the 4 field sample data points (25% of the total) that were assessed and qualified with a 'J' validation flag can be categorized as definitive data with a positively identified analyte concentration that is only an estimate due to low CCV, MS, MSD, and/or AS recovery.

The USEPA TCLP regulatory level for cadmium (1000ug Pb/L) and mercury (200ug Hg/L) was exceeded by BWXT reported cadmium (1,000ug Pb/L) and mercury (226ug Hg/L) results for sample 1RD01002TI. Therefore, according to USEPA regulations (See section 7.4.1. in Chapter 7 of SW-846), the waste represented by sample 1RD01002TI possesses the characteristic of toxicity.

**7.0 Flagrant Contractual Deficiencies**

**7.1 Missed Holding Times**

None

**7.2 Use of Unauthorized Methods**

None

**7.3 Other(s)**

**7.3.1. Laboratory Data Flags**

The laboratory failed to 'N' flag barium and cadmium results, as required by ERD-SOW-107R2, sec. 4.4.6, due to matrix spike and matrix spike duplicate results that were outside the acceptable control limits. These 'N' flags have been manually entered during validation; no further qualification of barium and cadmium results is warranted as a result of this action (CTR Comment #10).

The laboratory failed to correctly 'E' flag cadmium and silver results, as required by ERD-SOW-107R2, sec. 4.4.7, due to analytical spike results that were outside the acceptable control limits. These 'E' flags have been manually entered during validation; no further qualification of cadmium and silver results is warranted as a result of this action (CTR Comment #11).



## **Additional Radionuclide Analysis Data and Data Validation**



FORM I

## BBWI SAMPLE MANAGEMENT OFFICE

## RADIONUCLIDE ANALYSIS RESULTS

Date: August 8, 2001

Lab Name:

BWLVA

Case No.:

ER-SOW-380R2

Report No.:

107074

SDG No.:

1RD05001R9

| BBWI ID    | Lab ID     | Sample Matrix | Anal Type | Sample Value | Sample Uncer | Units | Anal Date | Sample Date | Sample Size | Yield | Det ID | MDA      |
|------------|------------|---------------|-----------|--------------|--------------|-------|-----------|-------------|-------------|-------|--------|----------|
| 1RD05001R9 | 0107074-01 | Sludge        | U-234(d)  | 1.36E+02     | 1.12E+01     | pCi/g | 08/03/01  | 04/30/01    | 1.0714      | 89.7  | A04    | 2.45E-01 |
| 1RD05001R9 | 0107074-01 | Sludge        | U-235(d)  | 6.88E+00     | 8.70E-01     | pCi/g | 08/03/01  | 04/30/01    | 1.0714      | 89.7  | A04    | 2.72E-01 |
| 1RD05001R9 | 0107074-01 | Sludge        | U-238(d)  | 7.52E-01     | 2.53E-01     | pCi/g | 08/03/01  | 04/30/01    | 1.0714      | 89.7  | A04    | 2.85E-01 |
| 1RD05002R9 | 0107074-02 | Sludge        | U-234(f)  | 1.31E+02     | 1.06E+01     | pCi/g | 08/03/01  | 04/30/01    | 1.0573      | 103.5 | A05    | 1.82E-01 |
| 1RD05002R9 | 0107074-02 | Sludge        | U-235(f)  | 6.78E+00     | 7.98E-01     | pCi/g | 08/03/01  | 04/30/01    | 1.0573      | 103.5 | A05    | 1.39E-01 |
| 1RD05002R9 | 0107074-02 | Sludge        | U-238(f)  | 9.65E-01     | 2.40E-01     | pCi/g | 08/03/01  | 04/30/01    | 1.0573      | 103.5 | A05    | 1.39E-01 |
| 1RD05101R9 | 0107074-03 | Sludge        | U-234(d)  | 1.01E+04     | 9.02E+02     | pCi/g | 08/03/01  | 04/30/01    | 0.0103      | 72.1  | A06    | 3.10E+01 |
| 1RD05101R9 | 0107074-03 | Sludge        | U-235(d)  | 4.09E+02     | 6.54E+01     | pCi/g | 08/03/01  | 04/30/01    | 0.0103      | 72.1  | A06    | 2.00E+01 |
| 1RD05101R9 | 0107074-03 | Sludge        | U-238(d)  | 1.80E+02     | 3.76E+01     | pCi/g | 08/03/01  | 04/30/01    | 0.0103      | 72.1  | A06    | 2.00E+01 |
| 1RD05102R9 | 0107074-04 | Sludge        | U-234(f)  | 7.87E+03     | 6.76E+02     | pCi/g | 08/03/01  | 04/30/01    | 0.0106      | 74.8  | A07    | 3.62E+01 |
| 1RD05102R9 | 0107074-04 | Sludge        | U-235(f)  | 3.22E+02     | 5.33E+01     | pCi/g | 08/03/01  | 04/30/01    | 0.0106      | 74.8  | A07    | 2.42E+01 |
| 1RD05102R9 | 0107074-04 | Sludge        | U-238(f)  | 1.48E+02     | 3.37E+01     | pCi/g | 08/03/01  | 04/30/01    | 0.0106      | 74.8  | A07    | 1.74E+01 |
| 1RD05201R9 | 0107074-05 | Sludge        | U-234(d)  | 7.08E+01     | 6.06E+00     | pCi/g | 08/03/01  | 05/01/01    | 1.1919      | 90.7  | A18    | 2.00E-01 |
| 1RD05201R9 | 0107074-05 | Sludge        | U-235(d)  | 3.85E+00     | 5.27E-01     | pCi/g | 08/03/01  | 05/01/01    | 1.1919      | 90.7  | A18    | 1.50E-01 |
| 1RD05201R9 | 0107074-05 | Sludge        | U-238(d)  | 5.27E-01     | 1.66E-01     | pCi/g | 08/03/01  | 05/01/01    | 1.1919      | 90.7  | A18    | 1.23E-01 |
| 1RD05202R9 | 0107074-06 | Sludge        | U-234(f)  | 7.48E+01     | 6.20E+00     | pCi/g | 08/03/01  | 05/01/01    | 1.0340      | 92.9  | A19    | 1.40E-01 |
| 1RD05202R9 | 0107074-06 | Sludge        | U-235(f)  | 4.95E+00     | 6.41E-01     | pCi/g | 08/03/01  | 05/01/01    | 1.0340      | 92.9  | A19    | 1.40E-01 |
| 1RD05202R9 | 0107074-06 | Sludge        | U-238(f)  | 9.49E-01     | 2.39E-01     | pCi/g | 08/03/01  | 05/01/01    | 1.0340      | 92.9  | A19    | 1.40E-01 |
| 1RD05301R9 | 0107074-07 | Sludge        | U-234(d)  | 3.08E+04     | 2.54E+03     | pCi/g | 08/03/01  | 05/02/01    | 0.0110      | 69.1  | A24    | 1.86E+01 |
| 1RD05301R9 | 0107074-07 | Sludge        | U-235(d)  | 3.74E+02     | 5.94E+01     | pCi/g | 08/03/01  | 05/02/01    | 0.0110      | 69.1  | A24    | 1.86E+01 |
| 1RD05302R9 | 0107074-08 | Sludge        | U-238(f)  | 3.01E+04     | 2.57E+03     | pCi/g | 08/03/01  | 05/02/01    | 0.0111      | 66.7  | A21    | 1.83E+01 |
| 1RD05302R9 | 0107074-08 | Sludge        | U-235(f)  | 1.15E+03     | 1.31E+02     | pCi/g | 08/03/01  | 05/02/01    | 0.0111      | 66.7  | A21    | 1.83E+01 |
| 1RD05302R9 | 0107074-08 | Sludge        | U-238(f)  | 3.18E+02     | 5.40E+01     | pCi/g | 08/03/01  | 05/02/01    | 0.0111      | 66.7  | A21    | 1.83E+01 |
| 1RD05401R9 | 0107074-09 | Sludge        | U-234(d)  | 2.40E+04     | 1.94E+03     | pCi/g | 08/03/01  | 05/03/01    | 0.0265      | 28.8  | A22    | 2.24E+01 |
| 1RD05401R9 | 0107074-09 | Sludge        | U-235(d)  | 8.45E+02     | 1.04E+02     | pCi/g | 08/03/01  | 05/03/01    | 0.0265      | 28.8  | A22    | 2.01E+01 |
| 1RD05402R9 | 0107074-10 | Sludge        | U-238(f)  | 2.33E+04     | 1.88E+03     | pCi/g | 08/03/01  | 05/03/01    | 0.0258      | 33.9  | A23    | 2.01E+01 |
| 1RD05402R9 | 0107074-10 | Sludge        | U-235(f)  | 1.08E+03     | 1.17E+02     | pCi/g | 08/03/01  | 05/03/01    | 0.0258      | 33.9  | A23    | 1.62E+01 |
| 1RD05402R9 | 0107074-10 | Sludge        | U-238(f)  | 7.99E+02     | 9.36E+01     | pCi/g | 08/03/01  | 05/03/01    | 0.0258      | 33.9  | A23    | 1.62E+01 |
| 1RD05501R9 | 0107074-11 | Sludge        | U-234(d)  | 5.29E+04     | 4.34E+03     | pCi/g | 08/03/01  | 05/08/01    | 0.0113      | 52.2  | A24    | 2.39E+01 |
| 1RD05501R9 | 0107074-11 | Sludge        | U-235(d)  | 2.35E+03     | 2.39E+02     | pCi/g | 08/03/01  | 05/08/01    | 0.0113      | 52.2  | A24    | 2.39E+01 |
| 1RD05501R9 | 0107074-11 | Sludge        | U-238(d)  | 9.61E+02     | 1.21E+02     | pCi/g | 08/03/01  | 05/08/01    | 0.0113      | 52.2  | A24    | 2.39E+01 |
| 1RD05502R9 | 0107074-12 | Sludge        | U-234(f)  | 5.65E+04     | 4.70E+03     | pCi/g | 08/03/01  | 05/08/01    | 0.0113      | 46.5  | A03    | 4.13E+01 |
| 1RD05502R9 | 0107074-12 | Sludge        | U-235(f)  | 2.52E+03     | 2.73E+02     | pCi/g | 08/03/01  | 05/08/01    | 0.0113      | 46.5  | A03    | 3.40E+01 |
| 1RD05502R9 | 0107074-12 | Sludge        | U-238(f)  | 1.06E+03     | 1.45E+02     | pCi/g | 08/03/01  | 05/08/01    | 0.0113      | 46.5  | A03    | 3.40E+01 |
| 1RD05601R9 | 0107074-13 | Sludge        | U-234(d)  | 5.67E+02     | 4.56E+01     | pCi/g | 08/03/01  | 05/03/01    | 1.2042      | 45.4  | A04    | 4.32E-01 |
| 1RD05601R9 | 0107074-13 | Sludge        | U-235(d)  | 2.80E+01     | 2.86E+00     | pCi/g | 08/03/01  | 05/03/01    | 1.2042      | 45.4  | A04    | 4.79E-01 |

## FORM I

## BBWI SAMPLE MANAGEMENT OFFICE

## RADIONUCLIDE ANALYSIS RESULTS

Date: August 8, 2001

Lab Name:

BWLVA

Case No.:

ER-SOW-380R2

Report No.:

107074

SDG No.:

1RD05001R9

| BBWI ID    | Lab ID     | Sample Matrix | Anal Type | Sample Value | Sample Uncer | Units | Anal Date | Sample Date | Sample Size | Yield | Det ID | MDA      |
|------------|------------|---------------|-----------|--------------|--------------|-------|-----------|-------------|-------------|-------|--------|----------|
| 1RD05601R9 | 0107074-13 | Sludge        | U-238(d)  | 1.31E+01     | 1.62E+00     | pCi/g | 08/03/01  | 05/03/01    | 1.2042      | 45.4  | A04    | 5.01E-01 |
| 1RD05602R9 | 0107074-14 | Sludge        | U-234(f)  | 5.61E+02     | 4.49E+01     | pCi/g | 08/03/01  | 05/03/01    | 1.2873      | 40.4  | A05    | 3.80E-01 |
| 1RD05602R9 | 0107074-14 | Sludge        | U-235(f)  | 3.05E+01     | 3.05E+00     | pCi/g | 08/03/01  | 05/03/01    | 1.2873      | 40.4  | A05    | 2.90E-01 |
| 1RD05602R9 | 0107074-14 | Sludge        | U-238(f)  | 1.33E+01     | 1.60E+00     | pCi/g | 08/03/01  | 05/03/01    | 1.2873      | 40.4  | A05    | 2.90E-01 |
| 1RD05701R9 | 0107074-15 | Sludge        | U-234(d)  | 4.50E+03     | 3.91E+02     | pCi/g | 08/03/01  | 05/03/01    | 0.0258      | 66.5  | A06    | 1.34E+01 |
| 1RD05701R9 | 0107074-15 | Sludge        | U-235(d)  | 1.84E+02     | 2.89E+01     | pCi/g | 08/03/01  | 05/03/01    | 0.0258      | 66.5  | A06    | 8.67E+00 |
| 1RD05701R9 | 0107074-15 | Sludge        | U-238(d)  | 3.06E+01     | 1.06E+01     | pCi/g | 08/03/01  | 05/03/01    | 0.0258      | 66.5  | A06    | 8.67E+00 |
| 1RD05702R9 | 0107074-16 | Sludge        | U-234(f)  | 3.93E+03     | 3.26E+02     | pCi/g | 08/03/01  | 05/03/01    | 0.0283      | 72.1  | A07    | 1.41E+01 |
| 1RD05702R9 | 0107074-16 | Sludge        | U-235(f)  | 1.56E+02     | 2.35E+01     | pCi/g | 08/03/01  | 05/03/01    | 0.0283      | 72.1  | A07    | 9.41E+00 |
| 1RD05702R9 | 0107074-16 | Sludge        | U-238(f)  | 2.75E+01     | 8.87E+00     | pCi/g | 08/03/01  | 05/03/01    | 0.0283      | 72.1  | A07    | 6.75E+00 |
| 1RD05801R9 | 0107074-17 | Sludge        | U-234(d)  | 3.43E+03     | 2.91E+02     | pCi/g | 08/03/01  | 05/07/01    | 0.0263      | 81.6  | A08    | 1.10E+01 |
| 1RD05801R9 | 0107074-17 | Sludge        | U-235(d)  | 1.11E+02     | 2.03E+01     | pCi/g | 08/03/01  | 05/07/01    | 0.0263      | 81.6  | A08    | 7.91E+00 |
| 1RD05801R9 | 0107074-17 | Sludge        | U-238(d)  | 7.34E+00     | 5.36E+00     | pCi/g | 08/03/01  | 05/07/01    | 0.0263      | 81.6  | A08    | 7.91E+00 |
| 1RD05802R9 | 0107074-18 | Sludge        | U-234(f)  | 3.49E+03     | 3.02E+02     | pCi/g | 08/03/01  | 05/07/01    | 0.0256      | 74.0  | A18    | 1.14E+01 |
| 1RD05802R9 | 0107074-18 | Sludge        | U-235(f)  | 1.19E+02     | 2.04E+01     | pCi/g | 08/03/01  | 05/07/01    | 0.0256      | 74.0  | A18    | 8.59E+00 |
| 1RD05802R9 | 0107074-18 | Sludge        | U-238(f)  | 1.05E+01     | 5.79E+00     | pCi/g | 08/03/01  | 05/07/01    | 0.0256      | 74.0  | A18    | 7.05E+00 |
| 1RD05901R9 | 0107074-19 | Sludge        | U-234(d)  | 5.98E+00     | 7.13E-01     | pCi/g | 08/03/01  | 04/30/01    | 1.4253      | 73.8  | A19    | 1.28E-01 |
| 1RD05901R9 | 0107074-19 | Sludge        | U-235(d)  | 2.02E-01     | 1.08E-01     | pCi/g | 08/03/01  | 04/30/01    | 1.4253      | 73.8  | A19    | 1.28E-01 |
| 1RD05901R9 | 0107074-19 | Sludge        | U-238(d)  | 2.49E+00     | 3.99E-01     | pCi/g | 08/03/01  | 04/30/01    | 1.4253      | 73.8  | A19    | 1.28E-01 |
| 1RD05902R9 | 0107074-20 | Sludge        | U-234(f)  | 5.29E+00     | 6.56E-01     | pCi/g | 08/03/01  | 04/30/01    | 1.5039      | 73.7  | A21    | 1.22E-01 |
| 1RD05902R9 | 0107074-20 | Sludge        | U-235(f)  | 3.07E-01     | 1.27E-01     | pCi/g | 08/03/01  | 04/30/01    | 1.5039      | 73.7  | A21    | 1.22E-01 |
| 1RD05902R9 | 0107074-20 | Sludge        | U-238(f)  | 3.11E+00     | 4.56E-01     | pCi/g | 08/03/01  | 04/30/01    | 1.5039      | 73.7  | A21    | 1.22E-01 |

Comments:

The Analysis Types have been annotated with a "f" or "d" designating the preparation technique used, where "f" represents a molten salt fusion method and "d" represents a strong mineral acid dissolution method.

## FORM II

## BWLVA SAMPLE MANAGEMENT OFFICE

## RADIONUCLIDE QUALITY CONTROL RESULTS

Lab Name:

BWLVA

Date:

August 8, 2001

Case No.:

ER-SOW-380R2

Report No.:

107074

SDG No.:

1RD05001R9

| QC Sample ID | Sample Type | Anal Type | Sample Value | Sample Uncer | Known Value | Known Uncer | Units | LCS Recov | Anal Date  | Chem Yield | Det ID | MDA      |
|--------------|-------------|-----------|--------------|--------------|-------------|-------------|-------|-----------|------------|------------|--------|----------|
| BL511-81     | BLK         | U-234     | 1.74E+01     | 5.16E+00     | 0.00E+00    | 0.00E+00    | pCi/L | NA        | 08/03/2001 | 78.7       | A23    | 3.61E+00 |
| BL511-81     | BLK         | U-235     | -3.35E-01    | 1.20E+00     | 0.00E+00    | 0.00E+00    | pCi/L | NA        | 08/03/2001 | 78.7       | A23    | 4.03E+00 |
| BL511-81     | BLK         | U-238     | 6.69E-01     | 1.52E+00     | 0.00E+00    | 0.00E+00    | pCi/L | NA        | 08/03/2001 | 78.7       | A23    | 3.61E+00 |
| 0107074-04D  | DUP         | U-234(f)  | 8.46E+03     | 7.33E+02     | 7.87E+03    | 6.76E+02    | pCi/g | NA        | 08/03/2001 | 84.1       | A08    | 2.65E+01 |
| 0107074-04D  | DUP         | U-235(f)  | 2.65E+02     | 4.87E+01     | 3.22E+02    | 5.33E+01    | pCi/g | NA        | 08/03/2001 | 84.1       | A08    | 1.90E+01 |
| 0107074-04D  | DUP         | U-238(f)  | 3.44E+02     | 5.71E+01     | 1.48E+02    | 3.37E+01    | pCi/g | NA        | 08/03/2001 | 84.1       | A08    | 1.90E+01 |
| QC511-81     | LCS         | U-234     | 6.20E+02     | 5.91E+01     | 4.83E+02    | 2.17E+01    | pCi/L | 128.3     | 08/03/2001 | 63.7       | A22    | 5.33E+00 |
| QC511-81     | LCS         | U-235     | 2.70E+01     | 7.41E+00     | 2.29E+01    | 1.01E+00    | pCi/L | 120.0     | 08/03/2001 | 63.7       | A22    | 4.78E+00 |
| QC511-81     | LCS         | U-238     | 5.81E+02     | 5.59E+01     | 4.83E+02    | 2.17E+01    | pCi/L | 120.2     | 08/03/2001 | 63.7       | A22    | 4.78E+00 |

Comments: The Analysis Types have been annotated with a "f" or "d" designating the preparation technique used, where "f" represents a molten salt fusion method

and "d" represents a strong mineral acid dissolution method.

**ORIGINAL**

**RADIOANALYTICAL DATA LIMITATIONS AND VALIDATION REPORT**

for the

**TANK V-9 (TSF-18) SAMPLING OU 1-10**

**RECEIVED**

**AUG 27 2001**

by

**ENVIRONMENTAL RESTORATION DEPT.**

**ENVIRONMENTAL DATA SERVICES, LTD.**

**Report Number  
ER354**

**Analyses Types  
Isotopic Uranium**

**SDG No.  
1RD05001R9**

Prepared by:

*Alexandra Washington*

Date:

*8/24/01*

Approved by:

*Diane Waldman*

Date:

*8.24.01*

## 1.

Twenty sludge samples were collected in April and May of 2001 to support the TANK V-9 (TSF-18) SAMPLING OU 1-10 program. The samples were analyzed for isotopic uranium at BWXT Services, Inc. – Nuclear Environmental Laboratory Services, Lynchburg, VA.

The laboratory data package met the Tier-1 reporting requirements as per ER-SOW-163. The data were validated in accordance with Level-A validation as defined in INEEL Guidance Document (GDE)-7003, "Levels for Analytical Methods Data Validation" and data validation technical procedure (TPR)-80 "Radiological Data Validation."

## 2.

A. L&V Report Number: ER354

B. SDG Number: 1RD05001R9

C. Number of Samples: 20

D. Sample Type/Matrix Sludge

E. Analyses Type: Isotopic Uranium

F. Reporting Tier: 1

G. Validation Level: A

H. TOS Number: ER-SOW-380R1 <sup>2</sup> *30m 8/21/01*

I. Analytical Laboratory: BWXT Services, Inc.-  
INEL Services

J. LTI Number: 0107074

K. Validator Affiliation: EDS Ltd.

L. Validator Name: Adrinna S. Washington

M. Completion Date: 08/24/01

**3.**

General precautions and limitations associated with radioanalytical method data validation apply to this L&V report and are described in Section 2 of TPR-80 (*Idaho National Engineering and Environmental Laboratory Sample Management Office Technical Procedure for Radioanalytical Data Validation*, TPR-80, Rev. 2, May 1997).

#### 4. SAMPLE IDENTIFICATION

The INEEL field sample identification numbers are listed on the INEEL chain-of-custodies included in this data package. Samples were collected in April and May 2001.

| FIELD SAMPLE ID<br>NUMBER | LABORATORY<br>ID NUMBER | SAMPLE<br>MATRIX | FIELD SAMPLE ID<br>NUMBER | LABORATORY<br>ID NUMBER | SAMPLE<br>MATRIX |
|---------------------------|-------------------------|------------------|---------------------------|-------------------------|------------------|
| 1RD05001R9                | 0107074-01              | SLUDGE           | 1RD05501R9                | 0107074-11              | SLUDGE           |
| 1RD05002R9                | 0107074-02              | SLUDGE           | 1RD05502R9                | 0107074-12              | SLUDGE           |
| 1RD05101R9                | 0107074-03              | SLUDGE           | 1RD05601R9                | 0107074-13              | SLUDGE           |
| 1RD05102R9                | 0107074-04              | SLUDGE           | 1RD05602R9                | 0107074-14              | SLUDGE           |
| 1RD05201R9                | 0107074-05              | SLUDGE           | 1RD05701R9                | 0107074-15              | SLUDGE           |
| 1RD05202R9                | 0107074-06              | SLUDGE           | 1RD05702R9                | 0107074-16              | SLUDGE           |
| 1RD05301R9                | 0107074-07              | SLUDGE           | 1RD05801R9                | 0107074-17              | SLUDGE           |
| 1RD05302R9                | 0107074-08              | SLUDGE           | 1RD05802R9                | 0107074-18              | SLUDGE           |
| 1RD05401R9                | 0107074-09              | SLUDGE           | 1RD05901R9                | 0107074-19              | SLUDGE           |
| 1RD05402R9                | 0107074-10              | SLUDGE           | 1RD05902R9                | 0107074-20              | SLUDGE           |

*1RD05901R9 and 1RD05902R9 are soils (MAPEP057) see BAM-104-01.*

#### 5. CONTRACT AND TECHNICAL REVIEW (CTR)

This section contains the contract and technical review comments that describe the findings and observations for each of the main verification and validation parameters described in TPR-80. The actions taken for each analysis and the reasons why a particular data qualifier flag was assigned are also included. The following verification and validation parameters were reviewed:

##### A. COMPLETENESS OF THE DATA REPORT PACKAGE

The BWXT data package was complete and did meet all the required Tier-1 reporting requirements.

##### B. EVALUATION OF REPORTED RESULTS

The radioanalytical results were properly reported and the reporting forms contained all the required sample and analytical information. The required MDA was not met for isotopic uranium in any samples in this data group. *This is most likely due to the activity level in the samples and the count time (shorter) needed for good counting statistics.*

##### C. DETECTOR SYSTEM CALIBRATIONS AND OPERATIONAL PERFORMANCE CHECKS

All calibrations, calibration verification checks, and background checks provided on ER-SOW-163 Form III's show that the instruments used were "in calibration" and operating properly during the counting/analysis of the reported samples.

##### D. LABORATORY CONTROL SAMPLE RESULTS

Laboratory control samples (LCS) were run with this set of data and each percent recovery satisfied the LCS acceptance criteria. The LCS acceptance criteria vary with the uncertainty (relative standard deviation) associated with the LCS result.

A laboratory control sample was processed for isotopic-U. All LCS recoveries for the uranium isotopes were outside of the acceptance tolerance window.

The samples that were observed to have statistically positive activity at the 95% confidence level for the uranium isotopes have been qualified as "J", estimated.



E. METHOD BLANK RESULTS

A laboratory-generated blank sample (method blank) analyzed with each sample delivery group is a means of determining the existence and magnitude of contamination resulting from the sample preparation and analysis/measurement process. Any statistically positive activity detected for a targeted radionuclide indicates a potential positive bias in the project sample result for that radionuclide.

Method blank BL511-81 was statistically positive at 2-Sigma TPU for U-234. The U-234 activity was also greater than the MDA. U-234 for method blank BL511-81 has been reported without qualification.

All samples in this data set were related to the U-234 method blank contamination. Those U-234 sample values were statistically positive at the 95% confidence level, but the mean difference values between the blank and samples were greater than three. Also, the sample results and the U-234 blank activity differ by a factor greater than ten. Therefore, the U-234 results required no qualification.

Nevertheless, the U-234 sample results were previously qualified for the related noncompliant laboratory control sample.

For all remaining radionuclides analyzed, the method blank results met the acceptance criteria (i.e., the results were not statistically positive and were less than their respective MDA's). No validation action was necessary.

F. DUPLICATE SAMPLE RESULTS

One laboratory-generated duplicate pair (split) was analyzed with this set of data for isotopic uranium. The laboratory demonstrated that duplicate precision for isotopic-U was achieved (i.e., the mean difference was  $\leq 3$  and/or the relative percent difference was  $\leq 30\%$  (solid).

Please note, mean difference and relative percent difference values were elevated for statistically positive isotopes only.

G. ANALYTICAL YIELDS

The efficiency of a radiochemical separation is determined and evaluated by measuring the analytical yield. A known amount of tracer or a chemical carrier added to the sample is used to determine chemical yield or recovery. The tracer employed, possesses chemical behavior similar to the target radionuclide. The tracer is an isotope, which is not expected occur in the sample to be analyzed and for most procedures, the recovery is determined using an isotope of the analyte of interest

All appropriate tracer yield values were present and evaluated. Upon review, all yields observed fell within the window of acceptance criteria.

H. HOLDING TIME

The holding time requirement (i.e., <6 months) was met.

I. SAMPLE PRESERVATION

Preservation for the sludge samples in this delivery group was not required.

J. INTERCOMPARISON QC RESULTS

Intercomparison QC results were provided by the laboratory for the EPA-Las Vegas Performance Evaluation Program, Mixed Analyte Performance Evaluation Program (MAPEP) – Department of Energy, and the Environmental Measurements Laboratory Department of Energy Quality Assessment Program. The laboratory demonstrates accuracy and precision for these analyses.

K. PERFORMANCE EVALUATION (PE) SAMPLES

There was no performance evaluation sample submitted with this sample delivery group.

**6. DATA LIMITATIONS AND USABILITY OVERVIEW**

This section provides an overview of the limitations of the data for each sample and for each analysis.

**6.1 Summary of Qualified Data**

The radionuclide analyses of the samples in this delivery group that received data qualifier flags are listed below.

**6.1.1 Isotopic Uranium Data**

Nearly all samples in this delivery group contained statistically positive activity at the 95% confidence level for isotopic uranium. Those uranium isotope sample results however, were assigned a "J" validation flag and qualified estimated, due to the related noncompliant laboratory control sample. The following describes the exceptions.

The U-238 values for samples IRD05801R9 and IRD05802R9 had no statistically positive activity at 2-Sigma TPU and have been flagged "U", nondetected.

Sample IRD05901R9 was found to exhibit no statistically positive activity at the 95% confidence level for U-235. The U-235 result has been flagged "U", nondetected.

## 6.2 Radioanalytical Data Quality Assessment Table

The data quality assessment table lists the quality-related findings of the verification and validation parameters for each analysis type.

Project Name: TANK V-9 (TSF-18) SAMPLING OU 1-10

L&V Report #: ER354 Validation Level: A Assessor's Affiliation: Env. Data Svcs.  
 SDG #: 1RD05001R9 Reporting Tier: 1 Assessor's Name: Adrianna Washington  
 TOS #: ER-TOS-380R/2 Samples by matrix: Sludge Assessment Date: 08/24/01  
 SOW #: ER-SOW-163 Laboratory Name: BWXT Services, Inc.-NEI

*done  
8/27/01*

| Analysis Type                         | Iso-U | Comment |   |
|---------------------------------------|-------|---------|---|
| <b>Verification Review Parameters</b> |       | Y       | N |
| 1. Data Package Completeness          | I     |         | X |
| 2. Evaluation of Reported Results     | Q     | X       |   |
| <b>Validation Review Parameters</b>   |       |         |   |
| 1. Instrument Calibration Checks      | I     |         | X |
| 2. Laboratory Control Samples         | Q     | X       |   |
| 3. Blank Samples                      | Q     | X       |   |
| 4. Duplicate Samples                  | I     |         | X |
| 5. Analytical Yields                  | I     |         | X |
| 6. Sample Holding Times               | I     |         | X |
| 7. Sample Preservation                | I     |         | X |
| 8. Intercomparison QC Results         | N/A   |         | X |
| 9. PE Sample Results (blinds)         | N/A   |         | X |

Quality Assessment Flags: I Parameter is in control (meets acceptance criteria). There are no problems with the sample results data.  
 Q Parameter is questionable. There may be minor problems with the sample results data.  
 O Parameter is out of control (does not meet acceptance criteria). There may be major problems with the sample results data.  
 NA Parameter is not applicable to this analysis.  
 Comments Flags: Y Yes indicates a comment was made and can be found on the Comment Sheet.  
 N No indicates no comment was made.

### 6.3 Data Qualifier (Validation Flag) Table

The data qualifier table lists the qualification (validation) flags assigned to each analysis result.

Project Name: TANK V-9 (TSF-18) SAMPLING OU 1-10

L&V Report #: ER354

Validation Level: A

Assessor's Affiliation: Env. Data Svcs.

SDG #: 1RD05001R9

Reporting Tier: 1

Assessor's Name: Adrianna Washington

TOS #: ER-SOW-380R12

Samples by matrix: Sludge

Assessment Date: 08/24/01

SOW #: ER-SOW-163

*8/27/01*

Laboratory Name: BWXT Services, Inc.-NEL

| Analysis Type | U-234 | U-235 | U-238 |
|---------------|-------|-------|-------|
| Sample Number |       |       |       |
| IRD05001R9    | J     | J     | J     |
| IRD05002R9    | J     | J     | J     |
| IRD05101R9    | J     | J     | J     |
| IRD05102R9    | J     | J     | J     |
| IRD05201R9    | J     | J     | J     |
| IRD05202R9    | J     | J     | J     |
| IRD05301R9    | J     | J     | J     |
| IRD05302R9    | J     | J     | J     |
| IRD05401R9    | J     | J     | J     |
| IRD05402R9    | J     | J     | J     |
| IRD05501R9    | J     | J     | J     |
| IRD05502R9    | J     | J     | J     |
| IRD05601R9    | J     | J     | J     |
| IRD05602R9    | J     | J     | J     |
| IRD05701R9    | J     | J     | J     |
| IRD05702R9    | J     | J     | J     |
| IRD05801R9    | J     | J     | U     |
| IRD05802R9    | J     | J     | U     |
| IRD05901R9    | J     | U     | J     |
| IRD05902R9    | J     | J     | J     |

MAPEP Soil See BAM-104-01.

MAPEP Soil

*Bdm*  
8/27/01

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DEFINITIONS OF DATA QUALIFIER (VALIDATION) FLAGS USED IN THIS REPORT

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| Flag | Definition                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       |
|------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| U    | <p>The analysis was performed, but no radioactivity was detected (i.e., the radioanalytical result was not statistically positive at the 95% confidence level and/or the result was below its MDA). The "U" qualifier flag is also applicable to any result reported as zero (0) (+/- an associated uncertainty).</p> <p>NOTE: <i>The radionuclide is not considered to be present in the sample</i></p>                                                                                                                                                                                                                                                                                                                                                                                                                                                         |
| UJ   | <p>The analysis was performed and a statistically positive result was reported at the 95% confidence interval. However, the result is highly questionable (false positive) due to analytical and/or laboratory quality control anomalies. The use of such a result is strongly discouraged. Analytical and quality control anomalies include such items as; significant blank contamination, known photopeak interferences and/or photopeak resolution problems, known matrix interferences, unacceptable laboratory control sample recoveries, serious instrument calibration problems, improper sample preservation, etc.</p> <p>NOTE: <i>The radionuclide may or may not be present in the sample and the result is considered highly questionable.</i></p>                                                                                                   |
| J    | <p>The analysis was performed and radioactivity was detected (i.e., the radioanalytical result is statistically positive at the 95% confidence level and is above its MDA). However, the result is questionable due to analytical and/or laboratory quality control anomalies and should, therefore, be used only as an estimated (approximated) quantity. Analytical and/or quality control anomalies include such items such as; laboratory duplicate imprecision, unsatisfactory analytical yields, insufficient laboratory control sample recoveries, unacceptable PE sample results, instrument calibration problems, improper sample preservation, etc.</p> <p>NOTE: <i>The radionuclide is considered to be present in the sample, but the result may not be an accurate representation of the amount of activity actually present in the sample.</i></p> |
| R    | <p>The analysis result is unusable and was rejected due to severe analytical and/or quality control problems.</p> <p>NOTE: <i>The radionuclide may or may not be present and the result is known to be inaccurate or imprecise.</i></p>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |

## Radioanalytical Data Quality Assessment Comment Sheet

Project Name: TANK V-9 (TSF-18) SAMPLING OU 1-10

**L&V Report: #354**

Laboratory Name BWXT Services, Inc.-NEI

Date: 08/24/01[illegible]

#### 6.4 Summary of Data Usability

There were a total of 60 radionuclide results associated with the samples in this sample delivery group (SDG). Fifty-seven of these results were assessed qualified with "J" validation flag. The remaining three results were qualified with a "U" validation flag.

The three results qualified with a "U" flag, are not statistically positive at the 95% confidence level and/or are below their MDA, and are therefore, considered nondetected (i.e., the radionuclide is not considered to be present in the samples). The 57 estimated or "J" flagged data cannot be categorized as definitive data. The use of these results is strongly discouraged.

#### 7. FLAGRANT CONTRACTUAL DEFICIENCIES

None.

#### 8. DEFINITIONS

The terminology, acronyms and definitions used in the L&V report are provided to assure that there is complete understanding of their application and use in the INEEL SMO data validation process.

*DOE/EML.* The U.S. Department of energy (DOE) Environmental Measurements Laboratory (EML) Quality Assessment Program (QAP).

*DOE/MAPEP.* The U.S. Department of Energy (DOE, Office of Environmental Management, Mixed Analyte Performance Evaluation Program (MAPEP), administered by the DOE-ID Radiological and Environmental Sciences Laboratory (RESL).

*Data Quality Assessment Flag.* Quality assessment flags are incorporated into a quality assessment table to provide information relating directly to the quality of the radioanalytical data. Quality assessment flags are not assigned to, nor are they associated with, individual project sample results. Further discussion can be found in Section 1.2 of this TPR-80.

*Data Qualifier Flag.* The flag (letter codes) assigned to individual sample results during the data validation process to indicate the potential limitations and usability of the sample data.

*Data Validation.* A systematic review and evaluation process, performed external from the data generator, that applies a defined set of performance-based criteria to a body of data that may result in physical qualification of the data. The purpose of data validation is to determine the quality and defensibility of the reported radioanalytical data (which provides a level of confidence that a radionuclide is present or absent), and to establish limitations, applications, and usability of the data.

*Difference Factor.* A mathematical test to determine the difference in activity levels between sample results and the method blank results. The equation is shown in Section 4.3.C.4 of TPR-80,

*EPAP/PEP.* The U.S. Environmental Protection Agency (EPA) Environmental Monitoring Systems Laboratory (EMSL) Performance Evaluation Studies Program (PESP).

*Laboratory Control Sample (LCS).* The LCS is a certified material or an aliquot of a matrix (blank), which is free of radionuclide interference's (and the constituents of interest), that is spiked with a known concentration of a target radionuclide(s) and is put through the entire analytical/measurement process. Provides an indication of the adequacy of the laboratory procedure to measure the constituent of interest.

*Laboratory Duplicate.* A laboratory-generated split of an actual sample that is put through the same exact analytical/measurement process as the original sample. Provides an indication of analytical variability/precision or sample inhomogeneity.

*Laboratory Task Identification (LTI).* This is the laboratory task identification (or work order) number assigned by the laboratory to the analytical data report package.

*Mean Difference (MD).* A standard statistical method of assessing differences between radioactivity measurements and determining the significance of those differences. It is used in this procedure to evaluate the statistical difference between method blank results and sample results and to evaluate results associated with duplicate measurements. The equation used to perform mean difference calculations is shown in Sections 4.3.C.4 and 4.4.C.3 of TPR-80.

*Method Blank.* A laboratory-generated sample, representative of the sample matrix being analyzed, that contains none of the constituents of interest that has gone through the entire analytical and measurement process using the same reagents added to the samples being analyzed. The blank provides verification that contamination has not occurred during the handling, preparation, and analysis of the samples.

*Minimum Detectable Activity (MDA).* The minimum amount of radioactivity that can be reliably detected in a sample (with an established degree of confidence) under certain defined sets of background, sample, instrument, analytical and measurement conditions. The MDA generally refers to a limit that is sample-specific and is determined from the actual sample being measured. It is more of an "at-the-moment" determination of what is actually detectable.

*Positive Value.* A statistical determination that identifies the "presence" of radioactivity in a sample when the analytical result is greater than two times the reported one sigma error of that result.

*Quality Assessment Flag.* Quality assessment flags are incorporated into a quality assessment table to provide information relating directly to the quality of the radioanalytical data. Quality assessment flags are not assigned to, nor are they associated with, individual project sample results. Further discussion can be found in Section 1.2 of this TPR-80.

*Relative Percent Difference (RPD).* A mathematical test used to determine the difference between sample results and duplicate results. The equation used to perform RPD calculations is shown in Section 4.4.C.4 of TPR-80.

*Statistically Positive.* A statistical determination that identifies the "presence" of radioactivity in a sample when the analytical result is greater than two times the reported one sigma error of that result.

*Yield.* Is a measure of the efficiency of the radiochemical separation process. It is determined by adding a known amount of radioactive tracer or chemical carrier to the sample prior to sample preparation and analysis and measuring the analytical yield (gravimetrically or radiometrically) at the completion of the analytical/measurement process. The yield determinations are used in the calculation of sample results.



**9. REFERENCES**

- A. Bechtel BWXT Idaho, LLC, "Radioanalytical Data Validation," Sample Management Office Technical Procedure, TPR-80, May 1997.
- B. Bechtel BWXT Idaho, LLC, "INEL Sample Management Office Statement of Work for Radionuclide Analysis," INEL-95/039, ER-SOW-163, February 1995.
- C. Bechtel BWXT Idaho, LLC, "Levels of Analytical Method Data Validation," Environmental Operations Branch Sample Management Office, GDE-7003, March 2001.

**10. ATTACHMENTS**

The following items are included as an attachment to this L&V report:

- A. The validated radionuclide analysis results (Forms I and II).
- B. The laboratory data package cover page and case narrative.
- C. A copy of the INEEL chain-of-custody form.
- D. The computations performed to assess sample duplicate results.
- E. ER-SOW-380R/2

*pan*  
*8/27/01*

